CHEMICAL REAGENTS

THEIR PURITY AND TESTS

A NEW AND IMPROVED TEXT
BASED ON AND REPLACING THE LATEST EDITION OF
KRAUCH
"DIE PRÜFUNG DER CHEMISCHEN REAGENTIEN AUF REINHEIT"

BY
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AUTHORIZED TRANSLATION
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DR. KRAUCH'S PREFACE

As long ago as the early seventies I felt the need of a treatise on Chemical Reagents, and thought of compiling such a work. I was then an assistant in the chemical laboratory of the Government Agricultural Experiment Station at Munster, and in my chemical work there met with constant difficulties on account of the great variations in the chemicals which were graded as "C. P." "puriss," etc. It was my aim to fix uniform standards for such chemicals as are used in analytical work, such standards to define closely the degree of purity of the chemicals and yet to be possible of attainment in practice by the manufacturer.

Not until 1888, however, did my work in this direction take the tangible form of a book, "*Die Pruefung der Chemischen Reagentien auf Reinheit.*" A second revised and enlarged edition was published in 1891. In 1896 a third edition, carefully revised and still further enlarged, was published. Since that time a good many changes have taken place, so that my book again needed revision. As I was unable to undertake this work, Mr. E. Merck, appreciating the usefulness of such a work as mine, published in 1905 what might be considered its up-to-date revision.

It is a source of gratification to me to see this work translated and placed before my colleagues across the sea. Such changes and additions as the translator has made in order to adapt the book to their particular needs, have my approval.

Darmstadt, February, 1907.

DR. C. KRAUCH.

TRANSLATOR'S PREFACE

THE desire of American chemists to rid themselves of the misleading term "C.P.," and to get in its stead definite statements as to the exact degree of purity of their reagents or the exact limit of the impurities, impelled the American Chemical Society, in 1902, to create a Committee on Purity of Reagents. In 1906 this Committee advised against the publication of a book, because — to use their own language — "until further work is done and more data collected, such a work would be practically only a duplicate of the work of Krauch."*

It is evident that the work of the Committee is being performed with most painstaking care, as, at the time of the last report, the Committee was working on "perfecting a method for the accurate colorimetric determination of traces of iron."* It is evident, too, that if the collection of data regarding other tests than those for iron is pursued with the same painstaking care, the complete report of the Committee will not be ready for publication for several years. My excuse for presenting this translation now lies, therefore, in the hope that, until the work of the Committee appears, chemists will generally make acceptance of their purchases conditional upon their coming up to the specifications of purity prescribed by this translation.

The additions mentioned by Dr. Krauch, in the preface he has kindly consented to write for this book, are limited to articles which are universally used here but do not seem to be so in Europe, and for which specifications could be found in authoritative American publications. They are Glacial Acetic Acid 99.5%, 36% Acetic Acid, Hydrochloric Acid of a specific gravity of 1.050, Nitric Acid with one of 1.40, Phosphoric Acid with one of 1.057, 10% Sulphuric Acid, 28% Ammonia Water, 95% Alcohol, and Glycerin of a 1.250 specific gravity. The slight changes obviously necessary in the text on account of these additions have been carefully made.

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* See Journal of the American Chemical Society, Vol. XXVIII, No. 8, pp. 61 and 62.

THE TRANSLATOR.

TRANSLATOR'S NOTE

For the sake of brevity, expressions throughout the book are sometimes used without full qualification. The following is then to be their interpretation.

Atomic Weights, Quantitative Calulations, etc., are based on the table of International Atomic Weights for 1906 in which O = 16.

Specific Gravities are given at + 15°C, compared with water at + 4°C.

Unweighable Residue or words to that effect apply to residues weighing 0.0005 gm. or less.

Solutions of chemicals to be tested are made from 1 gm. diluted to 20 cc. or proportionately.

Volumetric Determinations can of course be made with other solutions than the potassium hydroxide and hydrochloric acid solutions generally selected, provided they be equivalent. In such substitution, however, due regard must be given to the suitableness of the indicator.

Nitric Acid has a specific gravity of 1.153.

Hydrochloric Acid, one of 1.124.

Ammonia Water, that of 0.96.

The **Marsh Apparatus** should be set up with a 200 cc. generating flask.

The tests presuppose the use of Jena or some equally resistant **Glassware.**

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CHEMICAL REAGENTS

ACETONE

$(CH_3)_2CO$. Mol. Wt. 58.04.

A clear, colorless liquid, boiling at 56 to 57° C., with a specific gravity of 0.797.

TESTS OF PURITY

Residue on Evaporation. — 25 cc. of acetone on evaporation should leave no weighable residue.

Acids. — Acetone should not redden blue litmus paper.

Solubility in Water. — Acetone should be miscible with an equal volume of water, yielding a clear liquid.

Aldehydes. — On heating 10 cc. of acetone with 5 cc. of ammoniacal silver nitrate solution for fifteen minutes on a steam-bath, the mixture must not acquire a brown color.

Substances Oxidizable by Permanganate. — On adding one drop of a 1:1000 solution of potassium permanganate to 10 cc. of acetone, and maintaining a temperature of 15° C., the pink color should not be entirely discharged within fifteen minutes.

Water. — On mixing equal volumes of acetone and petroleum benzin (boiling-point 40° to 70° C.), two layers should not be formed.

Note. — Regarding the quantitative determination of acetone, see G. Lunge, Chem.-tech. Untersuch.-Meth., 4 ed., **3**, 653 (1900). G. Krämer, Ber., **13**, 1000 (1880); J. Chem. Soc., **38**, 826 (1880). J. Messinger, Ber., **21**, 3366 (1888); J. Chem. Soc., **56**, 313 (1889). H. Strache, Monatsh. Chem., **12**, 524 (1891) [or Ztschr. anal. Chem., **31**, 573 (1892)]; J. Chem. Soc., **62**, 546 (1892). F. Robineau and G. Rollin, Ztschr. anal. Chem., **33**, 87 (1894); J. Chem. Soc., **64**, II, 556 (1893).

ACID ACETIC

$HC_2H_3O_2$. Mol. Wt. 60.03.

I

ACID ACETIC, 99.5%

A clear, colorless liquid, with a strong odor; miscible in all proportions with water or alcohol, and containing at least 99.5 per cent of $HC_2H_3O_2$. Specific gravity about 1.048 at 25° C. At about + 15.65° C. it solidifies.

TESTS OF PURITY

Non-volatile Matter. — 10 cc. of acetic acid should leave no weighable residue on evaporation.

Hydrochloric Acid. — 5 cc. of acetic acid diluted with 50 cc. of water should afford no turbidity on the addition of 5 cc. of nitric acid followed by silver nitrate solution.

Sulphuric Acid. — On boiling a mixture of 10 cc. of acetic acid and 150 cc. of water, and then adding barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

Heavy Metals and Earths. —

(*a*) A mixture of 20 cc. of acetic acid and 100 cc. of water should not be affected by passing through it a current of hydrogen sulphide gas.

(*b*) On diluting 10 cc. of acetic acid with 100 cc. of water and adding an excess of ammonia water, no green color should be developed on the addition of ammonium sulphide solution; nor should a turbidity or precipitate form on adding ammonium oxalate solution.

Formic and Sulphurous Acids. — 2 cc. of the acid are supersaturated with 8 to 10 cc. of ammonia water and a little silver nitrate solution added. No dark deposit should occur on boiling two minutes.

Substances Reducing Permanganate. — 2 cc. of the acid are diluted with 10 cc. of water, and 0.1 cc. of decinormal solution of potassium permanganate added. The pink color should not be entirely changed to brown within two hours.

Other Empyreumatic Bodies. — No smoky odor should be noticeable upon making 10 cc. of the acid alkaline with solution of potassium hydroxide.

Quantitative Determinations. — Dilute 10 gm. of acetic acid with sufficient water to make 100 cc. Titrate 10 cc. of this solution with normal potassium hydroxide solution, using phenolphthalein as the indicator.

1 cc. of normal KOH = 0.06003 gm. $HC_2H_3O_2$, log. 77837.

The acetic acid content may also be ascertained from the specific gravity, if due note be taken of the fact that the specific gravities above 1.0553 represent in each case two liquids of different acetic acid content.*

II

ACID ACETIC, GLACIAL, 96%

A clear, colorless liquid with a pungent odor, solidifying at about + 10° C.; specific gravity 1.064, and boiling point 117 to 118° C. It contains at least 96 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

This acid should respond to the tests for non-volatile matter, hydrochloric acid, sulphuric acid, heavy metals, and quantitative determinations, as described under Acid Acetic 99.5 per cent. Its strength, however, is less. The following test also applies.

Substances Reducing Permanganate. — On adding 0.3 cc.

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* Compare G. Lunge, Chem.-tech. Untersuch.-Meth., 4 ed., **3**, 681 (1900). Also U. S. Pharmacopœia, VIII, p. 610 (1905).

of decinormal potassium permanganate solution to a mixture of 5 cc. of acetic acid with 15 cc. of water, the red color should not disappear within fifteen minutes.

III

ACID ACETIC, 90%

A colorless liquid having a specific gravity 1.071 and containing at least 90 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests to be made are those given under Acid Acetic, Glacial, 96 per cent, observing the conditions there described.

IV

ACID ACETIC, 36%

A clear, colorless liquid, specific gravity about 1.048, and containing about 36 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests of purity and details of execution are the same as those given under Acid Acetic, Glacial, 96 per cent. But instead of 10 gm. (or cc.) of 96 per cent, use 25 gm. (or cc.) of the 36 per cent acid.

V

ACID ACETIC, DILUTED, 30%

A colorless liquid having the specific gravity 1.041. The liquid contains about 30 per cent of $HC_2H_3O_2$.

TESTS OF PURITY

The tests to be made are those given under Acid Acetic, Glacial, 96 per cent, observing the conditions there described. But instead of 10 gm. of the 96 per cent, use 30 gm. of the 30 per cent.

(ACID) ACETIC ANHYDRIDE

$(CH_3 \cdot CO)_2O$. Mol. Wt. 102.05.

A colorless liquid of pungent odor, boiling at 137° C. Specific gravity 1.08.

On pouring acetic anhydride into water, it does not at first mix with the latter, but sinks to the bottom of the vessel. Gradually, however, it dissolves, forming acetic acid.

TESTS OF PURITY

Hydrochloric Acid. — 1 cc. of acetic anhydride diluted with 50 cc. of water, and acidulated with 5 cc. of nitric acid, should give no reaction with silver nitrate solution.

Non-volatile Matter. — 10 cc. of acetic anhydride should leave no weighable residue on evaporation.

Quantitative Determination. — Dissolve 10 gm. of acetic anhydride in water and dilute to 100 cc. Titrate 10 cc. of this solution with normal potassium hydroxide solution, using phenolphthalein as indicator. It should require at least 19.3 cc. of the normal potassium hydroxide solution to develop the pink color.

1 cc. of normal KOH = 0.051025 gm. of $(CH_3 \cdot CO)_2O$, log. 70777.

(ACID) BORIC ANHYDRIDE

(BORON TRIOXIDE; BORIC ACID, FUSED)

B_2O_3. Mol. Wt. 70.0.

Brittle, vitreous, hygroscopic lumps used in silicate analysis, according to Jannasch.*

TESTS OF PURITY

Silica, Alkalies, etc. — Add 50 cc. of methyl alcohol-hydrochloric acid * to 5 gm. of the powdered boric anhydride in a weighed platinum dish, stir with a platinum wire until completely dissolved, and evaporate the liquid over a small flame, the platinum dish being placed on wire gauze. If a residue remains, it is treated anew with 25 cc. of the methyl alcohol-hydrochloric acid, the liquid evaporated, and the dish and contents gently ignited. There must remain no weighable residue.

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* *References :* P. Jannasch and O. Heidenreich, Ztschr. anorgan. Chem., **12**, 211 (1896) [or Ztschr. anal. Chem., **36**, 382 (1897)]; J. Chem. Soc., **70**, II, 576 (1896).

ACID CARMINIC †

$C_{22}H_{22}O_{13}$

Purple-brown, amorphous masses, affording a dark red powder on trituration. Carminic acid is easily soluble in water and in alcohol, but is insoluble in benzene and chloroform. It has no constant melting-point.

TESTS OF PURITY

Solubility. — 1 gm. of carminic acid dissolves completely in 2 cc. of water. The addition of 20 cc. of 90 per cent alcohol to this solution should cause no appreciable precipitation.

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* The methyl alcohol-hydrochloric acid is prepared by saturating pure, anhydrous methyl alcohol, keeping the temperature low, with thoroughly dried hydrochloric acid gas. On standing for some time, however, the methyl alcohol-hydrochloric acid loses its original effectiveness.

† *References:* E. Schunck and L. Marchlewski, Ber., **27**, 2979 (1894); J. Chem. Soc., **68**, I, 67 (1895). C. Liebermann and H. Voswinckel, Ber., **30**, 688 (1897); J. Chem. Soc., **72**, I, 292 (1897). Ber., **30**, 1731 (1897); J. Chem. Soc., **72**, I, 539 (1897). C. Liebermann, P. Höring, and F. Wiedemann, Ber., **33**, 149 (1900); J. Chem. Soc., **78**, I, 236 (1900). J. Landau, Ber., **33**, 2446 (1900); J. Chem. Soc., **78**, I, 661 (1900). C. Liebermann and J. Landau, Ber., **34**, 2153 (1901); J. Chem. Soc., **80**, I, 545 (1901). C. Liebermann and S. Lindenbaum, Ber., **35**, 2910 (1902); J. Chem. Soc., **82**, I, 787 (1902).

Regarding the use of carminic acid as a reagent see T. J. Bogolomow and N. J. Wassilieff, Pharm. Centrbl., **40**, 120 (1899); E. Merck, Annual Report (on year 1898).

If the carminic acid is intended to be used as an indicator in acidimetry, it must first be tested as to its sensitiveness, as follows:

Dissolve 1 gm. of the carminic acid in 100 cc. of water and add one drop of this solution to a solution of 5 gm. of ammonium chloride in 50 cc. of water. The addition to this latter solution of one drop of decinormal potassium hydroxide solution should cause a change in color from yellowish-red to violet-red.

ACID CITRIC

$C_6H_8O_7 + H_2O$. Mol. Wt. 210.08.

Colorless, odorless, rhombic prisms, efflorescing superficially in warm air.

Citric acid is soluble in 0.75 part of cold and in 0.5 part of boiling water; in 1 part of 85 per cent alcohol, and in 50 parts of ether.

TESTS OF PURITY

Oxalic and Tartaric * Acids. — On dissolving 1 gm. of citric acid in 2 cc. of water, and adding 10 drops of a 1:2 potassium acetate solution and 5 cc. of alcohol 85 per cent, no turbidity should be produced, nor should a crystalline deposit form within two hours.

Tartaric Acid and Sugar. — 1 gm. of citric acid and 10 cc. of sulphuric acid are ground together in a porcelain mortar previously rinsed with sulphuric acid. When this mixture is then heated in a test tube for an hour in a boiling water-bath it acquires at most a slight yellow color, but no brown color should develop.

Sulphuric Acid. — 20 cc. of the 1:10 aqueous solution must not be rendered turbid by the addition of barium chloride solution.

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* Regarding the detection of tartaric acid in citric acid see Merck's Reagentien-Verzeichnis (1903), p. 170; A. I. Cohn, Tests and Reagents (1903), pp. 40, 51, 242.

Calcium. — 20 cc. of the 1:10 aqueous solution must not be rendered turbid by the addition of ammonium oxalate solution.

Lead.* — A solution of 5 gm. of citric acid in 10 cc. of water, to which 12 cc. of ammonia water are added, should not acquire a dark color upon adding freshly made, saturated hydrogen sulphide water.

Non-volatile Matter. — 1 gm. of citric acid must leave no weighable residue on ignition.

Quantitative Determination. — On dissolving 1 gm. of citric acid in 30 cc. of water and titrating with normal potassium hydroxide solution, using phenolphthalein as indicator, not less than 14.2 cc. of the alkali solution should be required to develop the pink color.

1 cc. of normal KOH = 0.07002 gm. of $C_6H_8O_7 + H_2O$, log. 84522.

ACID GALLIC

$C_6H_2(OH)_3 \cdot (COOH) + H_2O$. Mol. Wt. 188.06.

Colorless or slightly yellowish needles or prisms, which melt and slowly decompose at 220° C. Gallic acid dissolves in 130 parts of cold water, in 3 parts of boiling water, in 5 parts of 95 per cent alcohol, and in about 40 parts of ether.

TESTS OF PURITY

Solubility in Water. — 1 gm. of gallic acid must dissolve completely in 20 cc. of water on heating. The solution must be colorless or only faintly yellowish.

Water Content. — On drying 1 gm. of gallic acid at 100° C.

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* Regarding the examination of citric acid for lead compounds see M. Bucket, Ztschr. anal. Chem. **32**, 465 (1893); J. Chem. Soc., **64**, II, 557 (1893).

to constant weight, it should not lose more than 0.1 gm. in weight.

Sulphuric Acid. — On adding 1 cc. of hydrochloric acid and some barium chloride solution to a 1:50 aqueous solution of gallic acid, a precipitate of barium sulphate must not form within an hour.

Inorganic Matter. — 1 gm. of gallic acid must leave no weighable residue on ignition.

ACID HYDRIODIC

HI. Mol. Wt. 127.97.

I

ACID HYDRIODIC, SP. GR. 1.5

A clear, colorless liquid which, on exposure to light and air, rapidly becomes yellow to brown, due to the separation of iodine. Specific gravity 1.5. The liquid contains about 43 per cent of HI.

TESTS OF PURITY

Non-volatile Matter. — 5 gm. of hydriodic acid should leave no weighable residue on evaporation.

Sulphuric Acid. — On diluting 5 gm. of hydriodic acid with 50 cc. of water and adding barium chloride solution, no barium sulphate should precipitate on standing twelve hours.

Heavy Metals and Earths. — On diluting 10 gm. of hydriodic acid with 100 cc. of water and passing hydrogen sulphide gas into a portion of the solution, no colored precipitate should form; and, after adding an excess of ammonia water to another portion of the solution, neither ammonium sulphide nor ammonium oxalate solution should cause a visible change.

Hydrochloric and Hydrobromic Acids. — Dilute 1 gm. of hydriodic acid with 20 cc. of water, add an excess of silver nitrate solution followed by 30 cc. of ammonia water, shake well, and filter. On acidulating the filtrate with nitric acid, only a slight turbidity may develop, never a precipitate.*

Quantitative Determination. — Dilute 5 gm. of hydriodic acid with 50 cc. of water and titrate with normal potassium hydroxide solution, using methyl orange as indicator. At least 17 cc. of the normal alkali solution must be required to effect the change in color.

1 cc. of normal KOH = 0.12797 gm. of HI, log. 10710.

II

ACID HYDRIODIC, SP. GR. 1.70

A yellow or brownish liquid of specific gravity 1.70. The acid contains about 57 per cent of HI and is used for methoxyl determination, according to Zeisel.

TESTS OF PURITY

Tests for non-volatile impurities and sulphuric acid are made, observing the conditions given above. In titrating this acid, phenolphthalein is used as the indicator.

ACID HYDROBROMIC

HBr. Mol. Wt. 80.96.

A clear, colorless, or faintly yellowish liquid, of specific gravity 1.38. It contains about 40 per cent of HBr.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of hydrobromic acid on evaporation on the water-bath should leave no weighable residue.

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* A slight turbidity is permitted, because silver iodide is not absolutely insoluble in ammonia water.

Sulphuric Acid. — On diluting 5 gm. of hydrobromic acid with 50 cc. of water and adding barium chloride solution, a precipitate of barium sulphate should not form on standing twelve hours.

Heavy Metals. —

(*a*) Dilute 10 gm. of hydrobromic acid with 10 cc. of water and overlay with hydrogen sulphide water; neither a coloration nor a yellow ring should form at the contact-surfaces of the two liquids on standing one hour.

(*b*) Dilute 20 cc. of hydrobromic acid with 20 cc. of water, and add 30 cc. of ammonia water, followed by a few drops of ammonium sulphide and ammonium oxalate solutions; on standing two hours neither a brown coloration nor a precipitate should appear.

(*c*) Boil 5 gm. of hydrobromic acid with 25 cc. of water and 2 drops of nitric acid. No reddish color should occur on the addition of pôtassium sulphocyanate solution.

Hydrochloric Acid. — 2 or 3 drops of hydrobromic acid are diluted with 4 cc. of water, and then mixed with 15 cc. of decinormal silver nitrate solution. To this mixture add 6 cc. of ammonium carbonate solution (1 part of ammonium carbonate, 1 part of ammonia water, and 3 parts of water), shake for five minutes, and filter. The filtrate acidulated with nitric acid may exhibit at most a faint turbidity.

Hydriodic Acid. — To a mixture of 5 drops of hydrobromic acid, 5 cc. of water, and 10 cc. of ammonia water, add one drop of silver nitrate solution. The mixture after being shaken must remain clear, or at most may develop a turbidity insufficient to destroy entirely its transparency.

Besides the above test for hydriodic acid, the following may also be employed:

On adding 1 drop of ferric chloride solution to 5 cc. of hydrobromic acid, and shaking with 5 cc. of chloroform, the latter should not acquire a violet color.

Phosphorous and Phosphoric Acids. Heat 1 gm. of hydrobromic acid with 1 cc. of nitric acid to boiling, allow to cool, and then add 4 cc. of ammonia water followed by 1 drop of magnesium sulphate solution. No precipitate should form even on standing two hours.

Quantitative Determination. Dilute 5 gm. of hydrobromic acid with 50 cc. of water, and titrate with normal potassium hydroxide solution, using methyl orange as indicator. At least 24.6 cc. of the normal alkali solution should be required to effect the change in color.

1 cc. of normal KOH = 0.08096 gm. of HBr, log. 90827.

ACID HYDROCHLORIC

HCl. Mol. Wt. 36.45.

I

ACID HYDROCHLORIC. SP. GR. 1.19

(Fuming Hydrochloric Acid)

A clear, colorless liquid, fuming in the air, and of specific gravity 1.19. The liquid contains about 37 per cent of HCl.

TESTS OF PURITY

Sulphuric Acid. Evaporate 100 gm. of hydrochloric acid in a platinum dish to about 5 cc., dilute the residue with water, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Non-volatile Matter. 20 gm. of hydrochloric acid, when evaporated in a platinum dish on the water-bath, must leave no weighable residue.

Chlorine.* — Dilute 5 gm. of hydrochloric acid with 50 cc. of water and add zinc iodide-starch solution. The liquid must not develop a blue color within ten minutes.

Sulphurous Acid. — 50 cc. of water are colored blue by the addition of 1 drop of decinormal iodine solution and a few drops of starch solution. Then add a mixture containing 5 gm. of the hydrochloric acid to be tested and 50 cc. of water. The liquid must not be decolorized after being shaken.

Heavy Metals. —

(*a*) Dilute 20 gm. of hydrochloric acid with 200 cc. of water, warm the liquid to about 70° C., and pass into it hydrogen sulphide gas for twenty minutes. No precipitate should form within two hours.

(*b*) Dilute 20 gm. of hydrochloric acid with 200 cc. of water, and add 50 cc. of ammonia water, followed by a few drops of ammonium sulphide solution. The mixture must not acquire a dark color, nor should a precipitate form.

(*c*) Boil 5 gm. of hydrochloric acid with 20 cc. of water and 2 drops of nitric acid. After cooling, a reddish color should not occur on the addition of potassium sulphocyanate solution.

Calcium. — On diluting 20 cc. of hydrochloric acid with 20 cc. of water and adding 50 cc. of ammonia water, followed by a few cc. of ammonium oxalate solution, no precipitate should form on standing two hours.

Arsenic. — Add 0.1 gm. of potassium chlorate to 200 gm. of hydrochloric acid, and evaporate on the water-bath. A Marsh apparatus is started, using 20 gm. of arsenic-free, granulated zinc and dilute (1: 5) sulphuric acid; then the residue from the above evaporation is washed into the generating flask of the apparatus by the aid of dilute sulphuric acid. After the apparatus has been in action for one hour, there may be at most a very slight deposit in the reduction tube, but no distinctly visible arsenic mirror.

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* Regarding the testing for chlorine, see also Kupfferschläger, Bull. Soc. chim. Paris, (3), **2**, 134 (1889) [Ztschr. anal. Chem., **31**, 201 (1892)]; J. Chem. Soc., **58**, 289 (1890).

Quantitative Determination. The strength of the acid may be most simply ascertained from the specific gravity.*

On diluting 3 gm. of hydrochloric acid with 50 cc. of water, and titrating with normal potassium hydroxide solution, using methyl orange as indicator, not less than 30.5 cc. of the normal alkali should be required to effect the color change.

1 cc. of normal KOH = 0.036458 gm. of HCl, log. 56179.

II

ACID HYDROCHLORIC, SP. GR. 1.124

(Diluted Hydrochloric Acid)

A clear, colorless liquid, of specific gravity 1.124, and containing 25 per cent of HCl.

TESTS OF PURITY

The tests to be made are those given under Fuming Hydrochloric Acid. But, instead of using 10 gm. of fuming acid, use 15 gm. of the acid of sp. gr. 1.124.

Quantitative Determination. Dilute 5 gm. of the acid with 50 cc. of water, and titrate with normal potassium hydroxide solution, using methyl orange as indicator. At least 34.3 cc. of the normal alkali should be required for neutralization.

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* See the table by Lunge and Marchlewski, in Lunge's Chem.-techn. Untersuch.-Meth., 5 ed., **1**, 419 (1904); U. S. Pharmacopœia, VIII, p. 612 (1905). Compare J. Soc. Chem. Ind., **24**, 789 (1905).

III

ACID HYDROCHLORIC, SP. GR. 1.050

A clear, colorless liquid, of specific gravity about 1.050, and containing about 10 per cent of HCl.

TESTS OF PURITY

The tests to be made are those given under Fuming Hydrochloric Acid. But, instead of using 10 gm. of the fuming acid, use 35 gm. of the 10 per cent acid.

ACID HYDROFLUORIC

HF. Mol. Wt. 20.

A colorless, or almost colorless, liquid, containing 38 to 40 per cent of HF. It fumes in the air.

TESTS OF PURITY

In these tests use platinum containers as much as possible.

Non-volatile Matter. — 20 gm. of hydrofluoric acid evaporated in a platinum dish, and gently ignited, should leave a residue weighing not more than 0.001 gm.

Sulphuric Acid. — Evaporate 2 gm. of hydrofluoric acid in a platinum dish on the water-bath, take up the residue with 10 cc. of water, and to this solution add a few drops of nitric acid and some barium nitrate solution. An immediate turbidity must not occur; and even after some time the liquid should exhibit, at most, a slight opalescence.

Calcium. — Dilute 5 gm. of hydrofluoric acid with 50 cc. of water and add ammonia water in excess, followed by ammonium oxalate solution. An immediate turbidity should not occur.

Magnesium. — Dilute 5 gm. of hydrofluoric acid with 50 cc. of water, and add ammonia water until the liquid has an alkaline reaction; on now adding ammonium phosphate solution, no precipitate should form on standing three hours.

Heavy Metals. —

(*a*) Dilute 10 gm. of hydrofluoric acid with 40 cc. of water, warm the liquid, and saturate with hydrogen sulphide gas. Neither a yellow nor a dark-colored precipitate should form.

(*b*) Dilute 5 gm. of hydrofluoric acid with 50 cc. of water, make the solution alkaline with ammonia water, and add ammonium sulphide solution. A green coloration must not develop, nor should a precipitate form.

Hydrochloric Acid. — Dilute 2 gm. of hydrofluoric acid with 50 cc. of water and add a few drops of nitric acid and silver nitrate solution. The liquid may exhibit, at most, a faint opalescence.

Hydrosilicofluoric Acid. — On diluting 5 gm. of hydrofluoric acid with 20 cc. of water and adding 2 cc. of a cold, saturated potassium chloride solution, the further addition of 40 cc. of 85 per cent alcohol should cause no turbidity or precipitate.

Quantitative Determination. — Dilute 2 gm. of hydrofluoric acid with 50 cc. of water and titrate with normal potassium hydroxide solution, using phenolphthalein as indicator.

1 cc. of normal KOH = 0.020 gm. of HF, log. 30103.

NOTE. — Regarding the titration of hydrofluoric acid, see Katz, Chem. Ztg., **28**, 356, 387 (1904); J. Chem. Soc., **86**, II, 442 (1904).

ACID HYDROSILICOFLUORIC

(FLUOSILICIC OR SILICOFLUORIC ACID)

H_2SiF_6. Mol. Wt. 144.41.

A clear, colorless liquid having a specific gravity of about 1.06. This specific gravity corresponds to a content of 7.5 per cent of H_2SiF_6.

TESTS OF PURITY

Non-volatile Matter. — 5 gm. of hydrosilicofluoric acid evaporated in a platinum dish should leave no weighable residue.

Heavy Metals. — Dilute 5 gm. of hydrosilicofluoric acid with 10 cc. of water, add a few drops of hydrochloric acid and 10 cc. of hydrogen sulphide water. No visible change should take place.

Sulphuric Acid. — On diluting 5 gm. of hydrosilicofluoric acid with 10 cc. of water and adding a solution of barium-free strontium nitrate, no precipitate should form, even on standing twelve hours.

NOTE. — Regarding the determination of hydrosilicofluoric acid, see Katz, Chem. Ztg., **28**, 356, 387 (1904); J. Chem. Soc., **86**, II, 442 (1904).

ACID IODIC

HIO_3. Mol. Wt. 175.97.

Colorless, rhombic crystals, or white, crystalline powder, easily soluble (1:1) in water, but difficultly soluble in alcohol. The aqueous solution first reddens blue litmus paper and then bleaches it.

TESTS OF PURITY

Non-volatile Matter. — On heating 2 gm. of iodic acid no weighable residue should remain.

Solubility. — 1 gm. of iodic acid should dissolve completely in 1 cc. of water, and yield a colorless solution.

Quantitative Determination. — Dissolve 1 gm. of iodic acid in water and dilute to 100 cc. Dilute 10 cc. of this solution with 50 cc. of water, and add 2 gm. of potassium iodide and 5 cc. of dilute sulphuric acid. Titrate the liberated iodine with decinormal sodium thiosulphate solution, using starch solution as indicator.

1 cc. of decinormal $Na_2S_2O_3$ = 0.002933 gm. of HIO_3, log. 46731.

(ACID) IODIC ANHYDRIDE

(IODINE PENTOXIDE)

I_2O_5. Mol. Wt. 333.94.

A white, crystalline powder, soluble in water with the formation of the hydrated acid, HIO_3, but insoluble in absolute alcohol, ether, and carbon disulphide.

TESTS OF PURITY

The tests to be made are those given under Iodic Acid.

1 cc. of decinormal $Na_2S_2O_3$ = 0.00278283 gm. of I_2O_5, log. 4448.

ACID MOLYBDIC

H_2MoO_4. Mol. Wt. 162.01.

A white or slightly yellowish powder, containing about 85 per cent of MoO_3.

TESTS OF PURITY

Solubility in Ammonia Water; Heavy Metals. 2 gm. of molybdic acid should completely dissolve in a mixture of 10 cc. of water and 5 cc. of ammonia water (sp. gr. 0.91), yielding a clear solution. On the addition of hydrogen sulphide water to this liquid it acquires a slight yellow color, a green color or a precipitate should not, however, develop.

Phosphoric Acid. Quantitative Determination. As detailed under (Acid) Molybdic Anhydride, below.

(ACID) MOLYBDIC ANHYDRIDE

(MOLYBDIC ACID, 100%)

MoO_3. Mol. Wt. 144.

A slightly yellowish powder, often exhibiting a faintly bluish tint, due to the presence of other oxides of molybdenum. The preparation contains about 100 per cent of MoO_3, and is free from ammonia and nitric acid.

TESTS OF PURITY

Alkalies. — On heating 1 gm. of molybdic anhydride in a test tube, the particles adhering to the heated portion begin to fuse at a red heat, while at the upper, cooler portion of the tube a crystalline formation of sublimed anhydride may be observed. If the molybdic anhydride is contaminated with salts of the alkalies, the whole melts to a dark-colored mass. In such case the melting-point is low.

Ammonium Salts. — On boiling 1 gm. of molybdic anhydride with sodium hydroxide solution, there must be no liberation of ammonia (to be ascertained by means of moistened litmus paper).

Solubility in Ammonia Water; Heavy Metals. — 2 gm. of molybdic anhydride, when gently heated with a mixture of 10 cc. of water and 5 cc. of ammonia water (sp. gr. 0.91), should entirely dissolve, yielding a clear solution. The solution acquires a slight yellow color on adding hydrogen sulphide water. This color must not change within ten minutes nor should a precipitate form.

Phosphoric Acid. — Dissolve 10 gm. of molybdic anhydride in 25 cc. of water and 15 cc. of ammonia water (sp. gr. 0.91). The solution, mixed with 150 cc. of nitric acid, and allowed to stand for two hours at a temperature of about 40° C., must not contain a yellow precipitate.

Nitric Acid. — Shake 1 gm. of molybdic anhydride with 10 cc. of water, and add a small crystal of sodium chloride, followed by one drop of a 1:1000 solution of indigo; the blue color of the solution must not disappear on adding 10 cc. of concentrated sulphuric acid.

Quantitative Determination. — Dissolve 0.5 gm. of molybdic anhydride in a mixture of 50 cc. of water and 1 cc. of ammonia water (sp. gr. 0.91), with the aid of a gentle heat. Acidulate the solution with 5 cc. of acetic acid (sp. gr. 1.041), dilute with 200 cc. of water, heat to boiling, then add a solution of 1.5 gm. crystallized lead acetate in 20 cc. of water. Boil for several minutes with constant stirring, whereby the precipitate, at first milky, is rendered granular and easy to filter. Collect the precipitate on a filter, previously dried at 100° C. and weighed, and wash it with boiling water until the washings cease to afford a reaction with hydrogen sulphide water. Dry the precipitate to constant weight at 100° C. and then ignite a portion of it. The ignited residue has the composition $PbMoO_4$.

$PbMoO_4 \times 0.39247 = MoO_3$, log. 59380.

ACID NAPHTHYLAMINESULPHONIC

(Alphanaphthylaminesulphonic Acid; Naphthionic Acid)

$C_{10}H_6(NH_2)(SO_3H)1:4 + \frac{1}{2}H_2O$. Mol. Wt. 232.18.

A white powder, or small, lustrous, colorless needles (when crystallized from hot water), which carbonize but do not melt on being heated. Naphthylaminesulphonic acid is soluble in about 4000 parts of cold water, more readily in hot water, scarcely soluble in alcohol, and insoluble in ether. The solution in ammonia water exhibits a violet fluorescence.

Note. — Regarding the use of naphthylaminesulphonic acid for the detection and colorimetric determination of small quantities of nitrous acid, see E. Riegler, Ztschr. anal. Chem., **35**, 677 (1896) [or Merck's Reagentien-Verzeichnis (1903), p. 120]; J. Chem. Soc., **72**, II, 230 (1897). E. Riegler, Ztschr. anal Chem., **36**, 306 (1897); J. Chem. Soc., **72**, II, 385 (1897).

ACID NITRIC

HNO_3. Mol. Wt. 63.04.

I

ACID NITRIC, SP. GR. 1.40

A clear, colorless liquid, specific gravity 1.40 to 1.42, and containing about 68 per cent by weight of HNO_3.

TESTS OF PURITY

Non-volatile Matter. — 4 cc. of nitric acid on evaporation should leave no weighable residue.

Sulphuric Acid. — Dilute 4 cc. of nitric acid with 90 cc. of water, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Hydrogen-Halogen Acids.—4 cc. of nitric acid diluted with 90 cc. of water must not appear changed on the addition of silver nitrate solution.

Heavy Metals, and Earths. — Dilute 8 cc. of nitric acid with 80 cc. of water, and render slightly alkaline with ammonia water. On adding a few drops of ammonium sulphide and ammonium oxalate solutions, neither a dark color nor a turbidity should result.

Iodic Acid and Iodine. — On diluting 2 cc. of nitric acid with 10 cc. of water, adding a small piece of metallic zinc, and shaking with a small quantity of chloroform, the chloroform should not be colored violet.

Quantitative Determination. — Dilute 2 gm. of nitric acid with 50 cc. of water and titrate with normal potassium hydroxide solution, using methyl orange as indicator.

1 cc. of normal KOH = 0.06304 gm. of HNO_3, log. 79962.

II

ACID NITRIC, SP. GR. 1.30

A clear, colorless liquid, of specific gravity 1.30, and containing about 47 per cent of HNO_3.

TESTS OF PURITY

The tests to be made are those given under Acid Nitric sp. gr. 1.40, observing the conditions there described. But instead of 4 cc. of the acid sp. gr. 1.40, 7.5 cc. of the acid sp. gr. 1.30 are to be used.

III

ACID NITRIC, SP. GR. 1.20

A clear, colorless liquid, of specific gravity 1.20, and containing about 33 per cent of HNO_3.

TESTS OF PURITY

The tests to be made are those given under Acid Nitric sp. gr. 1.40, observing the conditions there described. But, instead of 4 cc. of acid sp. gr. 1.40, 10 cc. of acid sp. gr. 1.20 are to be used.

IV

ACID NITRIC, SP. GR. 1.153

A clear, colorless liquid, of specific gravity 1.153, and containing about 25 per cent of HNO_3.

TESTS OF PURITY

The tests to be made are those given under Acid Nitric sp. gr. 1.40, observing the conditions there described. But instead of 4 cc. of acid sp. gr. 1.40, 13 cc. of acid sp. gr. 1.153 are to be used.

ACID NITRIC, CRUDE, SP. GR. 1.38

A clear, colorless, or yellowish liquid of specific gravity 1.380–1.40, and containing at least 61 per cent of HNO_3.

TESTS OF PURITY

Non-volatile Matter. — 5 cc. of crude nitric acid on evaporation should leave no weighable residue.

ACID NITRIC, FUMING, SP. GR. 1.486

A yellow, or reddish-yellow, clear liquid of specific gravity 1.486–1.500, and containing at least 86 per cent of nitric acid, HNO_3.

TESTS OF PURITY

The tests to be made are those given under Acid Nitric sp. gr. 1.40. But for 4 cc. of acid sp. gr. 1.40, use 5 cc. of fuming nitric acid.

ACID OXALIC

$H_2C_2O_4 + 2H_2O$. Mol. Wt. 126.04.

Colorless, odorless, prismatic crystals, free from efflorescence. Crystallized oxalic acid is soluble in 10 parts of cold, and in about 3 parts of boiling, water, in 2.5 parts of alcohol, and in about 100 parts of ether. On heating the hydrated oxalic acid in a capillary tube, it liquefies at 98° C. in its water of crystallization. On being heated to 70° C., the hydrated acid is rendered anhydrous, which latter sublimes at about 100° C. and melts at 187° C. This preparation must contain 99.8–100 per cent of the hydrated acid, $H_2C_2O_4 + 2\,H_2O$.

TESTS OF PURITY

Ash. — 3 gm. of oxalic acid, after being dried and then ignited in a platinum crucible, should leave no weighable residue.

Sulphuric Acid. — Dissolve 5 gm. of oxalic acid in 100 cc. of water, and add 1 cc. of hydrochloric acid and 1 cc. of barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — Dissolve 5 gm. of oxalic acid in 50 cc. of water, add 15 cc. of nitric acid and a few drops of silver nitrate solution. The solution may exhibit at most a slight opalescent turbidity.

Heavy Metals. — The 1:10 aqueous solution must be perfectly clear, and on adding hydrogen sulphide water to 30 cc. of it, no reaction should be observed. On now adding ammonia water to this liquid until alkaline, neither a green nor a brown coloration should develop, nor should a precipitate form.

Ammonium Compounds. —

(*a*) The solution of 5 gm. of crystallized oxalic acid in 30 cc. of sodium hydroxide solution on being boiled should not evolve ammonia (to be ascertained by means of moistened litmus paper).

(*b*) On dissolving 2.5 gm. of oxalic acid and 5 gm. of potassium hydroxide in 30 cc. of water, and adding to the solution about 15 drops of Nessler's reagent, at most a slight yellow color should develop; never a brownish-red color.

Nitric Acid. — On overlaying 10 cc. of a solution of diphenylamine in concentrated sulphuric acid with 10 cc. of a 1:10 aqueous solution of oxalic acid, a blue zone should not form at the contact-surfaces of the two liquids.

Quantitative Determinations. —

(*a*) Acidimetrically: Dissolve about 6.3 gm. of crystallized oxalic acid in water, dilute to 500 cc., and titrate 25 cc. of the solution with one fifth normal potassium hydroxide solution, using phenolphthalein as indicator. Perform the titration at 60° C.

1 cc. of fifth normal KOH = 0.012605 gm. of $H_2C_2O_4 + 2H_2O$, log. 10055.

(*b*) By Oxidimetry: To 25 cc. of the aqueous solution (6.5 gm. of crystallized oxalic acid in water as above) add 6 to 8 cc. of concentrated sulphuric acid, heat to about 60° C., and titrate with decinormal potassium permanganate solution.

1 cc. of decinormal $KMnO_4$ = 0.0063024 gm. of $H_2C_2O_4 + 2H_2O$, log. 79950.

ACID OXALIC, SUBLIMED

$H_2C_2O_4$. Mol. Wt. 90.01.

A white, crystalline, exceedingly hygroscopic powder, melting at 187° C. The preparation must contain at least 99.5 per cent of anhydrous oxalic acid, $H_2C_2O_4$.

TESTS OF PURITY

Ash. — 10 gm. of the oxalic acid on ignition should leave no weighable residue.

Other Tests. — The other tests to be made are those given under crystallized oxalic acid.

1 cc. of fifth normal KOH = 0.009001 gm. of $H_2C_2O_4$, log. 95432.

1 cc. of decinormal $KMnO_4$ = 0.0045008 gm. of $H_2C_2O_4$, log. 65329.

NOTE. — If it is desired to use sublimed oxalic acid for standardizing solutions for alkalimetric or oxidimetric determinations, it must be heated to dryness at a temperature of 60 to 70° C., in small quantities at a time.

ACID PERCHLORIC

$HClO_4$. Mol. Wt. 100.45.

A colorless liquid of specific gravity 1.12, and containing about 20 per cent of $HClO_4$.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of perchloric acid when evaporated and ignited should leave no weighable residue.

Sulphuric Acid. — On diluting 5 cc. of perchloric acid with 100 cc. of water, and adding 1 cc. of hydrochloric acid, followed by barium chloride solution, a precipitate of barium sulphate should not form on standing twelve hours.

Hydrochloric Acid. — The mixture obtained by diluting 5 cc. of perchloric acid with 25 cc. of water and adding 3 cc.

of nitric acid should not be rendered more than slightly turbid by silver nitrate solution.

Barium. — 10 cc. of perchloric acid diluted with 50 cc. of water must not become turbid within five minutes on adding dilute sulphuric acid.

Heavy Metals. — 10 cc. of perchloric acid diluted with 40 cc. of water must show no change on adding hydrogen sulphide water; and on adding to this solution 10 cc. of ammonia water, and a few drops of ammonium sulphide solution, a precipitate should not form, nor should the liquid acquire a green or brown color.

ACID PHOSPHOMOLYBDIC

$12MoO_3 \cdot H_3PO_4 + xH_2O.$

Yellow, lustrous crystals, easily and completely soluble in water, and yielding with the latter an acid solution.

TESTS OF PURITY

Solubility, Heavy Metals, and Earths. — 1 gm. of phosphomolybdic acid should completely dissolve in 10 cc. of water. On adding to this solution two or three drops of ammonia water, a yellow precipitate forms, which completely redissolves on the addition of 5 cc. of the ammonia water. On now adding to this solution ammonium sulphide and ammonium oxalate solutions, no visible change should take place.

ACID PHOSPHORIC

(Ortho.)

H_3PO_4. Mo. Wt. 98.02.

I

ACID PHOSPHORIC, SP. GR. 1.7

A clear, colorless, odorless, syrupy liquid, of specific gravity 1.7, and containing about 85 per cent of H_3PO_4.

TESTS OF PURITY

Volatile Acids. — Mix 30 cc. of the phosphoric acid with 50 cc. of water in a distilling flask. Distil * off 50 cc., and titrate the distillate with decinormal potassium hydroxide solution, using methyl orange as indicator. Not more than 0.1 cc. of the decinormal alkali solution should be required.

Nitric Acid. — 2 cc. of phosphoric acid mixed with 2 cc. of concentrated sulphuric acid, and overlaid with 1 cc. of a solution of ferrous sulphate, must not develop a colored zone.

Hydrogen-Halogen Acids and Phosphorous Acid. — 2 cc. of phosphoric acid diluted with 18 cc. of water should give no reaction with silver nitrate solution either in the cold or on warming.

Sulphuric Acid. — On adding barium chloride solution to 20 cc. of the diluted acid (1:10), no precipitate of barium sulphate should form on standing two or three hours.

Metaphosphoric Acid. — On dropping the acid diluted with 10 volumes of water into a dilute solution of albumen, no turbidity should ensue.

Heavy Metals, Earths, Etc. —

(*a*) Dilute the acid with 10 volumes of water, and to 20 cc. of the mixture add hydrogen sulphide water. There should be no visible change.

(*b*) On adding 10 cc. of ammonia water to 20 cc. of the above diluted acid, no precipitate should form on the further addition of either ammonium oxalate solution, or ammonium sulphide solution.

(*c*) A mixture of 5 cc. of phosphoric acid with 20 cc. of absolute alcohol should remain perfectly clear.

Substances Oxidizable by Permanganate. — On adding 5 drops of decinormal potassium permanganate solution to a mixture of 5 cc. of phosphoric acid and 5 cc. of diluted sulphuric acid, and then heating for five minutes at 100° C., the red color of the liquid should not disappear.

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* In order to prevent spirting over of phosphoric acid, the flask should be provided with a Kjeldahl connecting bulb tube. The distillate must be tested with ammonium molybdate solution for phosphoric acid..

Arsenic. — A Marsh apparatus is started using 20 gm. of arsenic-free, granulated zinc, and diluted (1:5) sulphuric acid. A mixture of 3 cc. of phosphoric acid with 20 cc. of water is then introduced in small quantities at a time, the evolution of hydrogen being maintained for about two hours. A deposit of arsenic should not be visible in the reduction tube within these two hours.

Quantitative Determination. — Dilute 1 gm. of phosphoric acid with 30 cc. of water, and titrate with normal potassium hydroxide, using phenolphthalein as indicator.* At least 17 cc. of normal alkali must be added to produce the red color.

1 cc. of normal KOH = 0.04901 gm. of H_3PO_4, log. 69028.

II

ACID PHOSPHORIC, SP. GR. 1.12

A clear, colorless, odorless liquid of specific gravity **1.12**, and containing about 20 per cent of H_3PO_4.

TESTS OF PURITY

The tests to be made are those given under Acid Phosphoric sp. gr. 1.7. But for 1 cc. of the phosphoric acid sp. gr. 1.7, use 4 cc. of the phosphoric acid sp. gr. 1.12.

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* Methyl orange may be used instead of phenolphthalein. In this case, however, 1 cc. of the normal KOH = 0.09802 gm. of H_3PO_4, log. 99131. When titrating with phenolphthalein, twice as many cubic centimeters of the alkali solution are required as when using methyl orange.

III

ACID PHOSPHORIC, SP. GR. 1.057

A clear, colorless, odorless liquid of specific gravity about 1.057, and containing about 10 per cent of H_3PO_4.

TESTS OF PURITY

The tests to be made are those given under Acid Phosphoric sp. gr. 1.7. But for 1 cc. of the acid sp. gr. 1.7, use 10 cc. of the acid sp. gr. 1.057.

(ACID) PHOSPHORIC ANHYDRIDE

(PHOSPHORUS PENTOXIDE)

P_2O_5. Mol. Wt. 142.00.

A white, amorphous, odorless, bulky powder, which dissolves in water with a hissing noise, forming metaphosphoric acid. Phosphoric anhydride completely sublimes on being heated.

TEST OF PURITY

Arsenous Acid. — Introduce 1 gm. of phosphoric anhydride, in small portions at a time, into 20 cc. of water, and while warming the solution, pass into it a current of hydrogen sulphide gas. The liquid should not acquire a yellow color, nor should a yellow precipitate form.

ACID PHOSPHORIC, META

(GLACIAL PHOSPHORIC ACID)

HPO_3. Mol. Wt. 80.0.

Colorless, transparent, vitreous pieces or sticks, deliquescent in moist air, and melting to a clear viscid liquid on being warmed. Metaphosphoric acid is very easily soluble in water.

435 543.01
N07

TESTS OF PURITY

Nitric Acid. — On adding 2 cc. of concentrated sulphuric acid to a solution of 1 gm. of metaphosphoric acid in 2 cc. of water, and overlaying the mixture with 1 cc. of a solution of ferrous sulphate, no colored zone should form.

Sulphuric Acid. — The solution of 1 gm. of metaphosphoric acid in 20 cc. of water must not immediately be rendered turbid on the addition of 5 cc. of hydrochloric acid, followed by barium chloride solution.

Hydrogen-Halogen Acids. — Add 5 cc. of nitric acid to a solution of 1 gm. of metaphosphoric acid in 20 cc. of water. On the further addition of silver nitrate solution the liquid should appear unchanged.

Heavy Metals, Earths, Etc. —

(*a*) The solution of 1 gm. of metaphosphoric acid in 20 cc. of water must not be affected by hydrogen sulphide water.

(*b*) On adding 5 cc. of ammonia water to a solution of 1 gm. of metaphosphoric acid in 20 cc. of water, no precipitate should form on adding ammonium oxalate and ammonium sulphide solutions.

Arsenic. — On adding 5 cc. of stannous chloride solution to a solution of 1 gm. of metaphosphoric acid in 1 cc. of water, the mixture should not darken on standing one hour.

Substances Oxidizable by Permanganate. — Dissolve 1 gm. of metaphosphoric acid in 10 cc. of water, add 5 cc. of 16 per cent sulphuric acid and 0.1 cc. of decinormal potassium permanganate solution; then heat 5 minutes at 100° C. The red color of the mixture should not disappear.

ACID PHOSPHOTUNGSTIC

$(P_2O_5 \cdot 20WO_3 \cdot 11H_2O) + 16H_2O$. Mol. Wt. 5268.43.

Small, white, or slightly yellowish-green crystals, easily soluble in water, and free from ammonia and nitric acid.

TESTS OF PURITY

Nitrates. — Dissolve 1 gm. of phosphotungstic acid in 10 cc. of water, add a granule of sodium chloride and 1 drop of indigo solution (1:1000), followed by 10 cc. of concentrated sulphuric acid. The blue color of the mixture must not disappear within ten minutes.

Ammonium Salts. — On heating a solution of 1 gm. of phosphotungstic acid in 10 cc. of water, with 5 cc. of sodium hydroxide solution (sp. gr. 1.3), no ammonia should be evolved (to be ascertained by means of dampened litmus paper).

NOTE. — Regarding a method for the quantitative analysis of phosphotungstic acid, see F. Kehrmann, Ber., **20**, 1813 (1887); J. Chem. Soc., **52**, 777 (1887).

ACID PICRIC

(PICRONITRIC ACID; TRINITROPHENOL)

$C_6H_2(OH)(NO_2)_3$. Mol. Wt. 229.14.

Pale yellow, glistening crystals, melting at 122.5° C., soluble in about 90 parts of cold water and in about 30 parts of boiling water. The acid is readily soluble in alcohol, ether, and benzene.

TESTS OF PURITY

Resins; Substances Insoluble in Water. — 1 gm. of picric acid should dissolve completely and without turbidity in 100 cc. of water. On adding to the solution 1 or 2 drops of 16 per cent sulphuric acid, no precipitate should form on standing twelve hours. On subsequently filtering the liquid, no resin should remain on the filter.

Picrates of Potassium, Sodium, and Ammonium.* — 1 gm. of picric acid must completely dissolve in 20 cc. of benzene, affording a clear solution.

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* Picric acid cannot be tested for ammonia by warming with sodium hydroxide solution, as under such conditions picric acid itself decomposes with the evolution of ammonia.

Oxalic Acid. — On adding calcium chloride solution to a solution of 1 gm. of picric acid in 100 cc. of water, a precipitate of calcium oxalate should not form within two hours.

Free and Combined Sulphuric Acid. Evaporate a mixture of 2 gm. of picric acid and 10 cc. of nitric acid (sp. gr. 1.4) to dryness on the water-bath. Dissolve the residue in 100 cc. of boiling water with the addition of 5 cc. of nitric acid, allow to cool, then filter, and add barium nitrate solution to the filtrate. There should be no immediate turbidity.

Inorganic Matter (Ash). 1 gm. of picric acid cautiously incinerated in an open platinum dish should not leave a residue weighing more than 0.001 gm.

ACID ROSOLIC

(CORALLIN)

Brittle, amorphous, reddish-brown pieces, presenting a metallic reflection, and readily soluble in alcohol, but insoluble in water.

Rosolic acid is used as an indicator in the form of a solution of 0.5 gm. of the acid in a mixture of 50 cc. of 85 per cent alcohol and 50 cc. of water.

TESTS OF SENSITIVENESS

Add two or three drops of the above rosolic acid solution to 100 cc. of distilled water. On adding to the solution 0.05 cc. of decinormal potassium hydroxide, the pale-yellow color of the water should change to a rose-red, and on the further addition of 0.05 cc. of decinormal hydrochloric acid, the original color should be restored.

ACID SUCCINIC

$C_4H_6O_4$. Mol. Wt. 118.04.

Colorless, monoclinic prisms, soluble in 20 parts of cold and about 2 parts of boiling water; in 10 parts of alcohol and in 80 parts of ether. Succinic acid melts at 182° C., and at 235° C. it boils with the evolution of white acrid fumes, while it undergoes decomposition to a large extent into water and succinic anhydride.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of succinic acid heated in a platinum dish should volatilize and leave no weighable residue; nor should any charring occur.

Oxalic Acid. — The solution of 1 gm. of succinic acid in 20 cc. of water should not be affected by calcium chloride solution.

Tartaric Acid and Sulphates. — On adding potassium acetate and barium nitrate solutions to a solution of 1 gm. of succinic acid in 20 cc. of water, no precipitate should form on standing twelve hours.

Chlorides. — On adding 2 or 3 cc. of nitric acid to 20 cc. of the 1:20 aqueous solution of succinic acid, not more than a faint opalescent turbidity should develop on the addition of silver nitrate solution.

Ammonium Salts. — 1 gm. of succinic acid, on being heated with 10 cc. of sodium hydroxide solution (sp. gr. 1.3), should liberate no ammonia (to be ascertained with moist litmus paper).

Heavy Metals. — 1 gm. of succinic acid dissolved in 20 cc. of water should not be visibly changed by hydrogen sulphide water.

Quantitative Determination. — Dissolve 1 gm. of succinic acid in 50 cc. of water, and titrate with normal potassium hydroxide, using phenolphthalein as indicator.

1 cc. of normal KOH = 0.05902 gm. of $C_4H_6O_4$, log. 77100.

ACID SULPHANILIC

$C_6H_4(NH_2)(SO_3H)1:4 + 2H_2O$. Mol. Wt. 209.18.

Sulphanilic acid occurs in colorless, acicular, efflorescent crystals. It has no melting-point, but carbonizes on being heated to 280 to 300° C. The acid is difficultly soluble in cold water (about 150 parts), but more readily soluble in hot water; it is insoluble in alcohol, ether, and benzene.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of sulphanilic acid should leave no weighable residue on ignition.

Sulphuric Acid (Aniline Sulphate). — A solution of 1 gm. of sulphanilic acid in 25 cc. of boiling water should not change in appearance on adding a few drops of barium chloride solution.

Hydrochloric Acid (Aniline Hydrochloride). — On shaking 1 gm. of sulphanilic acid with 20 cc. of water and filtering, the filtrate should exhibit at most a faint opalescent turbidity on the addition of a few drops of nitric acid and silver nitrate solution.

ACID SULPHURIC

H_2SO_4. Mol. Wt. 98.07.

I

ACID SULPHURIC, SP. GR. 1.84

(CONCENTRATED SULPHURIC ACID)

A clear, colorless, oily liquid of specific gravity 1.84, and containing 95 to 96 per cent of H_2SO_4.

TESTS OF PURITY

Non-volatile Matter. — 10 cc. of sulphuric acid, on being evaporated and ignited, should leave no weighable residue.

Nitric Acid. — Dilute 1 cc. of sulphuric acid with 9 cc. of water, and overlay this liquid on 5 cc. of diphenylamine solution (see Diphenylamine, page 100). A blue zone should not form at the contact-surfaces of the two liquids.

Selenium. — On overlaying 2 cc. of sulphuric acid with 2 cc. of hydrochloric acid, in which a granule of sodium sulphite has been dissolved, a reddish zone must not form at the contact-surfaces of the two liquids; nor should a red precipitate form on warming.

Substances Oxidizable by Permanganate. (Nitrous and Sulphurous Acids). — Dilute 15 cc. of sulphuric acid with 60 cc. of water, and color the solution by adding 1 drop of decinormal potassium permanganate solution. The pink color should not disappear within three minutes.

Hydrogen-Halogen Acids. — 2 cc. of sulphuric acid diluted with 30 cc. of water should appear unchanged on adding a few drops of silver nitrate solution.

Lead. — On cautiously diluting 10 cc. of sulphuric acid with 50 cc. of 85 per cent alcohol, no turbidity should be observed; nor should a precipitate of lead sulphate form on standing two hours.

Heavy Metals and Calcium. —

(*a*) Dilute 10 cc. of sulphuric acid with 50 cc. of water, and add ammonia water in excess. On now adding a few drops of ammonium sulphide and ammonium oxalate solutions, neither a green color nor a turbidity should occur.

(*b*) On diluting 20 cc. of sulphuric acid with 100 cc. of water, and passing a current of hydrogen sulphide gas into the solution, no brown color should develop;

nor, after the passage of the gas, should any brown flocks precipitate on long standing (Tin).

Ammonium Salts. — Dilute 2 cc. of sulphuric acid with 30 cc. of water, and add aqueous solution of potassium hydroxide (1:6) to alkalinity, followed by 10 to 15 drops of Nessler's reagent. At most a light-yellow precipitate may form, but in no case a brownish-red one.

Arsenic.* — A Marsh apparatus is set in operation, using 20 gm. of arsenic-free, granulated zinc, and arsenic-free, dilute sulphuric acid (1:5). As soon as the air has been expelled from the apparatus, heat the reduction tube to redness, and then introduce a cooled mixture of 10 cc. of the acid (sp. gr. 1.84) to be tested and 50 cc. of water. No deposit of arsenic should be visible in the reduction tube within half an hour.

Quantitative Determination. — The content of H_2SO_4 is most readily ascertained by taking the specific gravity, and then referring to tables for the percentage. Consult the tables of Lunge, Isler, and Naef, Chem.-tech. Untersuch. Meth., 5 ed., **1**, 354 (1904); compare Watts' Dict. of Chem., **4**, 621 (1894); also J. Soc. Chem. Ind., **24**, 790 (1905).

The acid content may also be ascertained by titration with normal potassium hydroxide solution, using methyl orange as indicator.

1 cc. of normal KOH = 0.049038 gm. of H_2SO_4, log. 69053.

II

ACID SULPHURIC, DILUTED 16%

A colorless liquid of specific gravity 1.110 to 1.114, and containing 15.7 to 16.3 per cent of H_2SO_4.

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* Regarding the detection of arsenic in sulphuric acid, see Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **1**, 367 (1904); Detection and Determination of Arsenic. Reprint from J. Soc. Chem. Ind., 1901.

TESTS OF PURITY

The tests to be made are those given under Acid Sulphuric sp. gr. 1.84. But for 1 cc. of the acid sp. gr. 1.84 use 9 cc. of the acid sp. gr. 1.110.

III

ACID SULPHURIC, 10%

A colorless liquid of specific gravity about 1.070, and containing about 10 per cent of H_2SO_4.

TESTS OF PURITY

The tests to be made are those given under Acid Sulphuric, sp. gr. 1.84. But for 1 cc. of the acid sp. gr. 1.84, use 15 cc. of the acid sp. gr. 1.070.

(ACID) SULPHURIC ANHYDRIDE

(Sulphur Trioxide)

SO_3. Mol. Wt. 80.06.

Long, transparent, colorless prisms, which melt at 15° C. to a clear oily liquid, boiling at 46° C. On long keeping at a temperature below 25° C., the sulphuric anhydride polymerizes to a modification which forms long, silky, felted needles, which melt above 50° C., and which at a higher temperature become converted into the trioxide in vapor form, solidifying at 15° C., and boiling at 46° C.

Note. — Regarding the quantitative determination of sulphuric anhydride, see the statements given under Acid Sulphuric, Fuming. Also R. Rosenlecher, "*Zur Gehaltsbestimmung der rauchenden Schwefelsäure und des Schwefelsäure anhydrids,*" Ztschr. anal. Chem., **37**, 209 (1898); J. Chem. Soc., **74**. II, 404 (1898).

ACID SULPHURIC, FUMING

$xH_2SO_4 + ySO_3$.

I

ACID SULPHURIC, FUMING. FREE FROM NITROGEN

A colorless, oily liquid, fuming in the air, and containing 8 to 10 per cent of free SO_3 (*i.e.*, 83.1 to 83.5 per cent total SO_3).

TESTS OF PURITY

Non-volatile Matter. — 3 cc. of fuming sulphuric acid, on being evaporated and ignited, should leave no weighable residue.

Nitric Acid. — Dilute 1 cc. of fuming sulphuric with 9 cc. of water, and overlay the fluid on 5 cc. of diphenylamine solution (see Diphenylamine, page 100). No blue zone should form at the contact-surfaces of the two liquids.

Ammonium Salts. — Carefully add 2 cc. of fuming sulphuric acid, by drops, to 30 cc. of water, and add aqueous solution (1 : 6) of potassium hydroxide to alkalinity; on now adding 10 to 15 drops of Nessler's reagent, at most a pale-yellow, but in no case a brownish-red, color or precipitate may develop.

Halogens. — Add 1 cc. of fuming sulphuric acid, by drops, to 30 cc. of water; on now adding a few drops of silver nitrate solution, the liquid must not acquire more than a faint opalescent turbidity.

Lead. — On cautiously adding 10 cc. of fuming sulphuric acid, by drops, to 50 cc. of 85 per cent alcohol, a clear liquid should result, which should contain no precipitate of lead sulphate after standing two hours.

Arsenic. — A Marsh apparatus is set in operation, using 20 gm. of arsenic-free, granulated zinc, and arsenic-free, dilute (1:5) sulphuric acid. As soon as the air has been expelled from the apparatus, heat the reduction tube to redness, then run into the apparatus a cooled mixture of 10 cc. of the fuming sulphuric acid with 90 cc. of water. No deposit of arsenic should be visible in the reduction tube within half an hour.

Quantitative Determination.[*] — Weigh off accurately 1 gm. of fuming sulphuric acid in a Lunge-Rey stoppered bulb pipette,[†] and cautiously allow the acid to run into 10 cc. of water, contained in a porcelain dish. The contents of the dish are now rinsed into a measuring flask of 100 cc. capacity, and the flask then filled up with water to the mark. After vigorously shaking, 25 cc. of the liquid are titrated with fifth normal potassium hydroxide solution, using methyl orange as indicator.[‡]

1 cc. of fifth normal KOH = 0.008006 gm. of SO_3, log. 90342.

II

ACID SULPHURIC, FUMING[¶]

An oily liquid, sometimes slightly colored, and often not perfectly clear. The acid fumes in the air, contains from

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[*] In accurate determinations, the acidity due to sulphurous acid must be deducted from that determined by titration. Regarding this, see Lunge, Chem.-tech., Untersuch.-Meth., 5 ed., **1**, 395 (1904); J. Chem. Soc., **68**, II, 413 (1895).

[†] See Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **1**, 394 (1904); illustrated in Trade Catalogues of glassware, etc.

[‡] In order to ascertain the free SO_3 from the total SO_3 found by analysis, see the table in Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **1**, 399 (1904); compare Thorpe's Dict. of Appld. Chem., 2 ed., **3**, 711 (1895).

[¶] This acid, which contains traces of nitric acid, is considerably cheaper than the foregoing acid, which is perfectly free from nitrogen. It is well adapted for use in laboratories where fuming sulphuric acid is very frequently used for the Kjeldahl nitrogen determinations. For accurate nitrogen determinations, however, it will be necessary to determine the nitrogen content of the acid by a blank test carried out in the manner above described.

8 to 10 per cent of free SO_3, and is intended for nitrogen determinations by the Kjeldahl method.

TESTS OF PURITY

Nitrogen. — Dilute 30 cc. of the fuming sulphuric acid in a retort with 200 cc. of water, and when cold add nitrogen-free solution of sodium hydroxide (sp. gr. 1.3), until strongly alkaline. Then add 3 gm. of zinc dust, distil off about 50 cc., and collect the distillate in a U-tube receiver containing about 10 cc. of water and 2 to 3 cc. of fifth normal hydrochloric acid. The distillate is then titrated with fifth normal potassium hydroxide solution, using methyl orange as indicator. The ammonia should not have consumed more than 0.2 cc. of the fifth normal hydrochloric acid.

Quantitative Determination. The determination is carried out as described under Acid Sulphuric, Fuming, free from nitrogen.

ACID SULPHURIC, WITH PHOSPHORIC ANHYDRIDE

APPROXIMATELY 10, 15, OR 20% P_2O_5

Sulphuric acid containing phosphorus pentoxide. Approximately 10, 15, or 20 per cent of P_2O_5 are the usual strengths. The preparation is employed in Kjeldahl's nitrogen determination.

TESTS OF PURITY

Nitric Acid. — Dilute 1 cc. of the solution of phosphoric anhydride in sulphuric acid with 9 cc. of water, and overlay this fluid on 5 cc. of diphenylamine solution (see Diphenylamine, page 100). No blue zone should form at the contact-surfaces of the two liquids.

Ammonium Salts. — Dilute 2 cc. of the solution of phosphoric anhydride in sulphuric acid with 30 cc. of water, and add aqueous solution of potassium hydroxide (1:6) to alkalinity, followed by 10 to 15 drops of Nessler's reagent. At most only a faint yellow, but in no case a brownish-red, color or precipitate should form.

ACID SULPHURIC, FUMING, WITH PHOSPHORIC ANHYDRIDE

Approximately 5, 10, 15, 20, or 25% P_2O_5

Fuming sulphuric acid containing phosphorus pentoxide; the usual grades contain approximately 5, 10, 15, 20, or 25 per cent of P_2O_5. The preparation is used in Kjeldahl's nitrogen determination.

TEST OF PURITY

Nitrogen. — The nitrogen determination is carried out as described under Acid Sulphuric, Fuming. See footnote there.

ACID SULPHUROUS

SO_2 + Aq. Mol. Wt. 64.06.

A clear, colorless liquid of specific gravity 1.029 to 1.035. The acid first reddens blue litmus paper, and then bleaches it. The liquid contains about 6 per cent of SO_2.

TESTS OF PURITY

Non-volatile Matter. — 10 cc. of sulphurous acid, on being evaporated and ignited, should leave no weighable residue.

Quantitative Determination. — Weigh off 10 gm. of sulphurous acid in a measuring flask of 100 cc. capacity, and fill with boiled water up to the mark. Allow the fluid to run from a burette into 30 cc. of decinormal iodine solution, constantly shaken, until decoloration ensues. To effect this not more than 16.2 cc. of the acid solution should be required.

1 cc. of decinormal I = 0.003203 gm. of SO_2, log. 50556.

CUBES FOR GENERATING SULPHUROUS ACID

Cube-like pieces, containing at least 20 per cent of sulphurous acid.

TEST OF STRENGTH

Quantitative Determination. — Introduce 1 gm. of the finely powdered cubes and 5 gm. of anhydrous sodium carbonate into a graduated flask of 100 cc. capacity; add 50 cc. of water, boil the mixture for about ten minutes, allow to cool, fill the flask up to the mark, and filter. Run the filtrate from a burette into a constantly shaken mixture of 30 cc. of decinormal iodine solution and 10 cc. of hydrochloric acid, until complete decoloration ensues.

1 cc. of decinormal I = 0.003203 gm. of SO_2, log. 50556.

ACID TANNIC

(TANNIN)

$C_{14}H_{10}O_9$. Mol. Wt. 322.08.

A yellowish powder, or crystal-like, lustrous scales. Tannic acid is soluble in 5 parts of water and in 2 parts of 85 per cent alcohol, yielding a clear liquid acid to litmus paper. Tannic acid is also soluble in about 8 parts of glycerin but is almost insoluble in ether.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of tannin on ignition should not leave a residue exceeding 0.002 gm. in weight.

Sugar, Dextrin. — On mixing 10 cc. of an aqueous (1 : 5) solution of tannic acid with 10 cc. of 85 per cent alcohol, the mixture must remain clear for one hour; nor should a turbidity occur on the further addition of 5 cc. of ether.

Water. — On drying tannic acid at 100° C., it should not lose more than 12 per cent of its weight.

NOTE. — Regarding the quantitative determination of tannic acid, see Fresenius, Anleitung zur quantitativen Analyse, Vol. II, 619 [or Lunge, Chem.-tech. Untersuch.-Meth., 4 ed., **3**, 560 ff. (1900)]; Fresenius-Cohn Quantitative Analysis, Vol. II, 767 ff. (1904).

ACID TARTARIC

$C_4H_6O_6$. Mol. Wt. 150.04.

Colorless, prismatic crystals, or crystalline crusts, soluble in 0.8 part of water, and in 2.5 parts of 85 per cent alcohol.

TESTS OF PURITY

Sulphuric and Oxalic Acids, and Calcium. — Separate 20 cc. portions of the aqueous (1:10) solution of the acid should show no change with barium chloride nor ammonium oxalate solutions; nor, when lowered to slight acidity by adding ammonia water, should a precipitate be obtained on adding calcium sulphate solution.

Lead and other Metals. —

(*a*) The solution of 5 gm. of tartaric acid in 20 cc. of water, with 12 cc. of ammonia water added, should not develop a brown color on the addition of hydrogen sulphide water.

(*b*) 20 cc. of the 1:10 aqueous solution should not be affected by hydrogen sulphide water.

Inorganic Matter.—1 gm. of tartaric acid, on being ignited, should leave no weighable residue.

Quantitative Determination. — Dissolve 1 gm. of tartaric acid in 50 cc. of water, and titrate with normal sodium hydroxide solution, using phenolphthalein as indicator.

1 cc. of normal NaOH = 0.07502 gm. of $C_4H_6O_6$, log. 87518.

ACID THIOACETIC

CH_3COSH. Mol. Wt. 76.09.

A yellow liquid of penetrating odor, boiling between 92 and 97° C., and having a specific gravity of 1.070. Thioacetic acid is soluble in 16 parts of water, and easily in alcohol. The 6 per cent aqueous solution is used instead of hydrogen sulphide in chemical analysis.*

TESTS OF PURITY

Non-volatile Matter. — 10 cc. of thioacetic acid, on being evaporated and ignited, should leave no weighable residue.

Sulphuric Acid. — On dissolving 5 cc. of thioacetic acid in 100 cc. of water, and adding barium chloride solution, no turbidity should occur, nor should a precipitate form.

ALCOHOL ETHYLIC

$C_2H_5 \cdot OH$. Mol. Wt. 46.04.

I

ALCOHOL ABSOLUTE

A clear, colorless liquid of specific gravity 0.796 to 0.798 (99 to 99.6 per cent by weight), and boiling at 78.5° C. Alcohol should not affect litmus paper.

TESTS OF PURITY

Residue. — 50 cc. of alcohol, on being slowly evaporated, should leave no residue.

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* Regarding the use of thioacetic acid, see the following: R. Schiff and N. P. Tarugi, Ber., **27**, 3437 (1894) [or Ztschr. anal. Chem., **34**, 456 (1895)]; J. Chem. Soc., **68**, II, 84 (1895). R. Schiff, Ber., **28**, 1204 (1895); J. Chem. Soc., **68**, II, 370 (1895).

Fusel Oil.* — On mixing 10 cc. of alcohol and 30 cc. of water in an Erlenmeyer flask, no turbidity or coloration should be observed, nor should any foreign odor be noticeable.

A mixture of 10 cc. of alcohol and 0.20 cc. of 15 per cent sodium hydroxide solution, when evaporated down to 1 cc. and supersaturated with dilute sulphuric acid, should not have the odor of fusel oil.

On rubbing a few drops of alcohol between the hands, no unpleasant odor should be noticeable.

Molasses-Alcohol. — On overlaying 5 cc. of alcohol on 5 cc. of concentrated sulphuric acid, no rose-red zone should form within one hour at the contact-surfaces of the two liquids.

Aldehyde. — On warming 10 cc. of alcohol with 5 drops of silver nitrate solution and 1 cc. of water for ten minutes on the water-bath (70 to 80° C.), neither a turbidity nor a precipitate should form.

Organic Impurities.† — The red color of a mixture of 10 cc. of alcohol and 1 drop of a 1:1000 potassium permanganate solution should not pass into yellow within twenty minutes.

Metals and Tannin. — On adding to 10 cc. of alcohol 1 cc. of ammonia water or 5 cc. of hydrogen sulphide water, no coloration should develop.

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* Regarding the quantitative determination of fusel oil, see Lunge, Chem.-tech. Untersuch.-Meth., 4 ed., **3**, 422, 442 (1900); Allen, Com. Organ. Anal., 3 ed., Vol. I, p. 167 ff. (1898). A. Stutzer and O. Reitmaier, Ztschr. angew. Chem., **3**, 522 (1890); J. Chem. Soc., **60**, 622 (1891). G. Lunge, V. Meyer, and E. Schulze, Chem. Centralb. (3), **15**, 854 (1884); J. Chem. Soc., **48**, 708 (1885). M. Glasenapp, Ztschr. angew. Chem., **8**, 657 (1895); J. Chem. Soc., **70**, II, 277 (1896). H. Bornträger, Chem. Ztg., **13**, Rep. p. 27 (1889); compare J. Chem. Soc., **56**, 552 (1889), and **58**, 669 (1890). See also Ztschr. Spiritusind., 1886, 362.

† A partial decolorization of potassium permanganate occurs even with the purest alcohol. Compare also M. E. Barbet, J. Pharm. Chim., **19**, 413, 457 (1889) [or Pharm. Ztg., **34**, 481 (1889)]. P. Cazeneuve, Chem. Ztg., **13**, Rep. p. 198 (1889); for similar article see J. Chem. Soc., **56**, 928 (1889). Lang, Chem. Ztg., **17**, 1544 (1893).

II

ALCOHOL, 95%

A clear, colorless liquid of about 0.816 specific gravity (15.6° C.), and containing about 95 per cent by volume of absolute alcohol, C_2H_5OH. It boils at 78° C., is miscible in all proportions with water, and does not change the color of moistened litmus paper.

TESTS OF PURITY

Residue. — 50 cc. of alcohol, on being slowly evaporated, should leave no weighable residue.

Fusel Oil. — Mix 10 cc. of alcohol, 5 cc. of water, and 1 cc. of glycerin, saturate a piece of clean, odorless blotting-paper with the mixture, and allow to evaporate spontaneously. No foreign odor should be noticeable after the evaporation.

Aldehyde, Tannin. — 10 cc. of alcohol mixed with 5 cc. of potassium hydroxide solution (5%) should not become yellow at once.

Foreign Organic Matter, Aldehyde, etc. Shake 20 cc. of alcohol with 1 cc. of decinormal silver nitrate solution. Not more than a faint opalescence should appear, nor more than a faint brownish tint after exposing for six hours to diffused daylight.

Furfural. — Add ten drops of aniline and two or three drops of hydrochloric acid to 10 cc. of alcohol. No pinkish-red color should appear.

III

ALCOHOL, 85%

This alcohol has a specific gravity of 0.830 to 0.834, and contains 87.2 to 85.6 per cent of alcohol, C_2H_5OH, by weight. In other respects it conforms to the requirements given under Alcohol Absolute.

ALCOHOL AMYLIC

$C_5H_{11} \cdot OH$. Mol. Wt. 88.07.

I

AMYL ALCOHOL

A clear, colorless liquid, neutral to litmus paper. Amyl alcohol is but slightly soluble in water, but is clearly miscible with alcohol, ether, and benzin. It has a specific gravity of 0.814 and boils at 131° C.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of amyl alcohol evaporated on the water-bath should leave no weighable residue.

Foreign Organic Matter (Furfural, etc.). —

(*a*) On shaking 5 cc. of amyl alcohol with 5 cc. of concentrated sulphuric acid, the mixture should not acquire more than a faint yellow or reddish color.

(*b*) On shaking 5 cc. of amyl alcohol with 5 cc. of potassium hydroxide solution, the amyl alcohol should not acquire any color.

II

AMYL ALCOHOL FOR GERBER'S FAT DETERMINATION

A colorless liquid boiling at 128 to 130° C., and of specific gravity 0.815.

TEST OF PURITY

1 cc. of amyl alcohol shaken with 10 cc. of concentrated sulphuric acid, and 11 cc. of water in a Gerber butyrometer, then centrifuged for two or three minutes, and then allowed to stand for twenty-four hours, must not exhibit an oily portion.*

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* Compare N. Gerber and M. M. Craandijk, Milch Ztg., **27**, 611 (1898). Chem. Centralb., [5], **2**, II, 907 (1898).

ALCOHOL METHYLIC

$CH_3 \cdot OH$. Mol. Wt. 32.03.

A colorless, mobile liquid, readily miscible in all proportions with water, alcohol, ether, and fatty and volatile oils. Methyl alcohol has the specific gravity 0.796, and boils between 65 and 66° C. It should not redden blue litmus paper.

TESTS OF PURITY

Non-volatile Matter. 30 cc. of methyl alcohol evaporated on the water-bath should leave no weighable residue.

Acetone and Ethyl Alcohol. Shake together thoroughly 50 cc. of double normal sodium hydroxide solution and 5 cc. of methyl alcohol in a mixing cylinder, and add, with repeated shaking, 25 cc. of double normal iodine solution. No turbidity or flocculent precipitate should occur; nor should an odor of iodoform be perceptible.

Empyreumatic Substances. 1 cc. of methyl alcohol should dissolve without turbidity in 10 cc. of water. On allowing 5 cc. of concentrated sulphuric acid to drop into 5 cc. of methyl alcohol while kept cold, the mixture should not acquire more than a slightly yellowish color.

Aldehydes.—On shaking 10 cc. of methyl alcohol with 10 cc. of sodium hydroxide solution (sp. gr. 1.3), the mixture should remain colorless.

Substances Oxidizable by Permanganate. On adding 1 drop of decinormal potassium permanganate solution to 10 cc. of methyl alcohol, the red color of the liquid must not disappear within ten minutes.

NOTE.—Regarding the quantitative determination of methyl alcohol, as well as the quantitative determination of acetone in methyl alcohol, see Lunge, Chem.-tech. Untersuch.-Meth., 4 ed., **3**, 693 (1900); compare G. Krämer, H. Strache, and J. Messinger as given in note under Acetone, p. 1.

ALPHANAPHTHOL

$C_{10}H_7 \cdot OH$. Mol. Wt. 144.06.

Colorless, lustrous needles, having a phenolic odor, and melting at 95° C. Alphanaphthol is difficultly soluble in cold water, more readily soluble in warm water, and dissolves easily in alcohol, ether, benzene, and chloroform.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of alphanaphthol ignited on platinum foil should leave no weighable residue.

Organic Acids. — On shaking 1 gm. of alphanaphthol with 100 cc. of water and filtering, the filtrate should not redden blue litmus paper.

ALUMINUM OXIDE

Al_2O_3. Mol. Wt. 102.2.

A white, very bulky, hygroscopic powder,* used for Wislicenus' † determination of tannin. Under a microscope, magnifying about 30 to 60 diameters, the powder appears in the form of partly transparent, flocculent masses, resembling vegetable tissue.

TESTS OF PURITY

Metallic Mercury and Aluminum. — On moistening the preparation with water or alcohol, it acquires a gray color if any aluminum or mercury is present; if pure, it remains perfectly white. No globules of mercury or particles of metallic aluminum should be visible under the microscope.

Absorptive Power for Tannin. — 1 gm. of freshly ignited aluminum oxide is vigorously shaken for five minutes with 200 cc. of a solution of 4.5 gm. of pure tannin in sufficient water to make 1000 cc. (Use a flask with a rubber stopper.) The mixture is then heated for five minutes on the water-bath, and filtered. 50 cc. of the filtrate are evaporated in a weighed platinum or nickel dish, and the residue dried at 105° C. and weighed.

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* The preparation must always be ignited before using in a determination of tannin.

† H. Wislicenus, Determination of Tannin Without Hide Powder, Ztschr. angew. Chem., **17**, 801 (1904); J. Soc. Chem. Ind., **23**, 765 (1904). Ztschr. anal. Chem., **44**, 96 (1905); J. Chem. Soc., **88**, II, 363 (1905).

The total residue obtained from 50 cc. of the original tannin solution, and dried at 105° C., is also ascertained. On now calculating the residue for 200 cc. of tannin solution, and deducting this from the total residue left from the tannin solution treated with the aluminum oxide, the result should show that 1 gm. of aluminum oxide absorbs 0.40 to 0.50 gm. of tannin.

AMMONIA WATER

$NH_3 + H_2O$. Mol. Wt. $NH_3 = 17.06$

The aqueous solution of ammonia gas is a clear, colorless liquid, the specific gravity of which decreases as the ammonia content increases.

For analytical purposes, two solutions of different concentration are ordinarily used, of which one has a specific gravity of about 0.96, and contains about 10 per cent of NH_3, while the other, the concentrated, has the specific gravity of about 0.925, and contains about 20 per cent of NH_3. In American laboratories a still more concentrated solution of ammonia is employed. Its strength is about 28 per cent and it is generally designated as "Stronger Ammonia Water."

I

AMMONIA WATER, 28%

(Stronger Ammonia Water)

A clear, colorless liquid having a specific gravity of about 0.90, and containing about 28 per cent of NH_3.

TESTS OF PURITY

Non-volatile Matter. — On evaporating 3.5 cc. of the ammonia water on the water-bath, no weighable residue should remain. A trace of residue, however, is almost always left.

Chlorides; Pyridine. — Dilute 3.5 cc. of the ammonia water with 30 cc. of water, and supersaturate with 20 cc. of nitric acid. The solution should remain colorless. This liquid should show no change on the addition of silver nitrate solution.

Heavy Metals. — On diluting 2 cc. of the ammonia water with 20 cc. of water, and then adding a few drops of ammonium sulphide solution, no change should appear.

Sulphates. — Slightly acidify 3.5 cc. of the ammonia water with hydrochloric acid, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Carbon Dioxide. — 3.5 cc. of the ammonia water with 15 cc. of calcium hydroxide solution should not at once become more than slightly opalescent.

Quantitative Determination. — Dilute 2 gm. of the ammonia water with about 50 cc. of water, and titrate with normal hydrochloric acid, using methyl orange indicator.

1 cc. of normal HCl = 0.01706 gm. of NH_3, log. 23198.

II

AMMONIA WATER, 20%

(CONCENTRATED AMMONIA WATER)

This solution of ammonia has the specific gravity 0.925 and contains about 20 per cent of NH_3.

TESTS OF PURITY

The tests for non-volatile matter, chlorides, pyridine, heavy metals, sulphates, and per cent of NH_3 as described under

Ammonia Water 28 per cent, are to be made. But, instead of 3.5 cc. of the 28 per cent use 5 cc. of the 20 per cent ammonia. The preparation should also conform to the following tests.

Sulphides.—On adding a few drops of an ammoniacal lead acetate solution to 5 cc. of the ammonia water, the liquid must not acquire a yellow or a brown color, nor should a dark precipitate form.

Tar-Bases (Aniline, Pyridine, Pyrrol, etc.).—Evaporate a mixture of 5 cc. of the ammonia water and 20 cc. of nitric acid on the water-bath. The residue on evaporation should have a pure white color.

Calcium.—A mixture of 5 cc. of the ammonia water with 20 cc. of water should exhibit no turbidity on the addition of ammonium oxalate solution.

Magnesium.—On adding ammonium phosphate solution to 10 cc. of the ammonia water, no precipitate should form on standing two hours.

Carbonates.—10 cc. of the ammonia water, when mixed with 20 cc. of lime water and boiled, should not develop more than a very slight turbidity.

Phosphates.—To 10 cc. of the ammonia water add 40 cc. of nitric acid and 25 cc. of ammonium molybdate solution. No yellow precipitate should form in the liquid on standing two hours at about 40° C.

III

AMMONIA WATER, 10%

This is the article termed "ammonia water" throughout the text of this book. It contains about 10 per cent of NH_3, and has a specific gravity about 0.96.

TESTS OF PURITY

The tests of purity and details of execution are the same as those given under Ammonia Water, 20 per cent. But, in testing this use double the quantities of ammonia water employed in testing the 20 per cent.

AMMONIUM ACETATE

$NH_4C_2H_3O_2$. Mol. Wt. 77.1.

A white, hygroscopic, crystalline mass, easily soluble in water and in alcohol.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium acetate, when ignited, should leave no weighable residue.

Chlorides. — The solution of 1 gm. of ammonium acetate in 20 cc. of water, acidulated with 5 cc. of nitric acid, should show no change on the addition of silver nitrate solution.

Sulphates. — On adding 1 cc. of hydrochloric acid to a solution of 1 gm. of ammonium acetate in 20 cc. of water, followed by barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

Heavy Metals and Earths. — The solution of 5 gm. of ammonium acetate in 100 cc. of water should not be affected by hydrogen sulphide water. Furthermore, the addition of ammonia water and ammonium oxalate solution should cause neither a coloration nor a turbidity.

AMMONIUM CARBONATE

$(NH_4)HCO_3 \cdot (NH_4)NH_2CO_2$.

The term "ammonium carbonate" is generally applied to a mixture of ammonium bicarbonate and carbamate, forming a crystalline, white, translucent mass which readily effloresces, becomes opaque, and gives off an odor of ammonia.

TESTS OF PURITY

Non-volatile Matter. — 5 gm. of ammonium carbonate, when ignited, should leave no weighable residue.

Sulphates. — Dissolve 5 gm. of ammonium carbonate in 100 cc. of water and 10 cc. of hydrochloric acid, heat the solution to boiling, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides and Thiosulphates. — The solution of 2 gm. of ammonium carbonate in 50 cc. of water should not be affected on adding 10 cc. of nitric acid, followed by silver nitrate solution.

Heavy Metals. — To a solution of 5 gm. of ammonium carbonate in 30 cc. of water is added 30 cc. of dilute acetic acid, then 20 cc. of ammonia water, then a few drops of ammonium sulphide solution. No precipitate should form, nor should the liquid acquire a green or a brown color.

Sulphocyanates. — The solution of 1 gm. of ammonium carbonate in 20 cc. of water and 2 cc. of hydrochloric acid should not be reddened on adding 1 drop of ferric chloride solution.

Tar Bases. — Acidify 1 gm. of ammonium carbonate with 5 cc. of nitric acid, and evaporate the solution to dryness on the water-bath. The residue must have a pure white color.

AMMONIUM CHLORIDE

NH_4Cl. Mol. Wt. 53.52.

A white, crystalline powder, easily soluble in water.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium chloride, when gently ignited, should leave no weighable residue.

Phosphates and Arsenates. — Dissolve 5 gm. of ammonium chloride in 20 cc. of water, and add to the clear solution 3 cc. of magnesia mixture and 10 cc. of ammonia water. No precipitate should form on standing twelve hours.

Heavy Metals and Earths. — 20 cc. portions of the aqueous 1:20 solution of the salt should not be affected by hydrogen sulphide water, ammonia water, ammonium sulphide solution, and ammonium oxalate solution.

Sulphates. — On adding to 10 cc. of the 1:10 solution a few drops of hydrochloric acid, followed by barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

Sulphocyanates. — Dissolve 1 gm. of ammonium chloride in 10 cc. of water, and add to the solution a few drops of hydrochloric acid and 1 drop of ferric chloride solution. The liquid should not acquire a red color.

Tar Bases. — On evaporating to dryness 1 gm. of ammonium chloride with 5 cc. of nitric acid on the water-bath, the residue must have a pure white color.

AMMONIUM CITRATE SOLUTION

A clear, colorless liquid, containing 150 gm. of pure, crystallized citric acid and 23 gm. of ammonia-nitrogen (= 27.93 gm. of NH_3) per liter. The solution has an acid reaction to litmus paper and is used for determining citrate-soluble phosphoric acid in Thomas slag, by the method of Wagner.

TEST FOR PROPER CONTENT OF AMMONIA-NITROGEN

Dilute 25 cc. of the ammonium citrate solution with water to 250 cc. To 25 cc. of this diluted solution add 3 gm. of calcined magnesia and about 200 cc. of water, and distil, using a receiver containing 40 cc. of semi-normal sulphuric acid. After the distillation titrate the excess of acid with semi-normal potassium hydroxide solution, using methyl orange as indicator.

1 cc. of semi-normal H_2SO_4 = 0.00702 gm. of N (log. 8463) = 0.00853 gm. of NH_3, log. 9309.

AMMONIUM DITHIOCARBONATE SOLUTION

$CO(SNH_4)_2$. Mol. Wt. 128.26.

A yellow liquid of ammoniacal odor, and containing about 10 to 12 per cent of ammonium dithiocarbonate, about 8 per cent of ammonium chloride, and small quantities of ammonium sulphocyanate and ammonium sulphide. It is used as a substitute for hydrogen sulphide and ammonium sulphide.

TESTS OF PURITY

Non-volatile Matter. — 10 cc. of ammonium dithiocarbonate solution, on being evaporated and ignited, should leave no weighable residue.

Ammonium Carbonate. — On adding 3 cc. of calcium chloride solution to 10 cc. of the ammonium dithiocarbonate solution, no precipitate should form, even on warming.

NOTE. — Regarding the employment of ammonium dithiocarbonate, see M. Vogtherr, Ber. d. pharm. Ges., **8**, 232 (1898) [or Pharm. Centrhl., **39**, 692 (1898)]; J. Chem. Soc., **78**, II, 241 (1900).

AMMONIUM FLUORIDE

NH_4F. Mol. Wt. 37.07.

White crystals, easily soluble in water, affording a solution which usually exhibits an acid reaction because of the presence of $NH_4F \cdot HF$.

TESTS OF PURITY

Non-volatile Matter. — 10 gm. of ammonium fluoride on ignition should leave no weighable residue.

Chlorides. — On dissolving 5 gm. of ammonium fluoride in

25 cc. of water, and adding a few drops of nitric acid, followed by silver nitrate solution, the liquid must exhibit no change.

Sulphates and Silicofluorides. — Dissolve 5 gm. of ammonium fluoride in a platinum dish in 25 cc. of water, and add 25 cc. of hydrochloric acid, followed by barium chloride solution. No turbidity should ensue.

Heavy Metals. — Dissolve 5 gm. of ammonium fluoride in 25 cc. of water, add to the solution a few drops of hydrochloric acid, and 10 cc. of hydrogen sulphide water. No change should appear. Now add ammonia water to alkalinity, and a few drops of ammonium sulphide solution; at most a slight greenish coloration may develop, but a precipitate should not form.

AMMONIUM MOLYBDATE

$(NH_4)_6Mo_7O_{24} + 4H_2O$. Mol. Wt. 1236.43.

Large, colorless, or slightly greenish crystals, soluble in water, which, when heated, liberate ammonia and water, leaving molybdic anhydride.

TESTS OF PURITY

Phosphates. — 10 gm. of ammonium molybdate with 25 cc. of water and 15 cc. of ammonia water (sp. gr. 0.910) should afford a clear solution. Add this solution to 150 cc. of nitric acid (sp. gr. 1.20 *). No yellow precipitate should form on standing two hours at a temperature of about 40° C.

Heavy Metals. — Dissolve 2 gm. of ammonium molybdate in 5 cc. of water and 5 cc. of ammonia water, and to the solution add 10 cc. of hydrogen sulphide water. Neither a green coloration nor a precipitate should form.

Sulphates. — On dissolving 1 gm. of ammonium molybdate in 10 cc. of water, and acidifying the solution with nitric acid, no change should be observed on the further addition of barium nitrate solution.

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* The ammonium molybdate solution should be added to the nitric acid gradually and with constant rotation; never otherwise.

Chlorides. — 20 cc. of the 1:10 aqueous solution acidified with nitric acid should show no change on the addition of silver nitrate solution.

Quantitative Determination. — The content of molybdic anhydride may be approximately determined by gently igniting about 1 gm. of the salt until the ammoniacal odor has disappeared, and then weighing the residue of molybdic anhydride; it should amount to about 81 per cent. The accurate determination is carried out in the manner described under Acid Molybdic Anhydride, on page 19.

AMMONIUM NITRATE

NH_4NO_3. Mol. Wt. 80.11.

Colorless crystals easily soluble in water.

TESTS OF PURITY

The tests to be made are those described under ammonium chloride; and, in addition, tests for chlorides and nitrites.

Chlorides. — On dissolving 1 gm. of ammonium nitrate in 10 cc. of water, and adding a few drops of nitric acid, followed by silver nitrate solution, the liquid should remain unchanged.

Nitrites. — To the solution of 1 gm. of ammonium nitrate in 20 cc. of water are added 1 cc. of 16 per cent sulphuric acid and 1 cc. of a freshly prepared (0.5:100*) colorless solution of metaphenylenediamine hydrochloride; no yellow or yellowish-brown color should develop.

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[*] Should the solution of metaphenylenediamine hydrochloride already have a color it is to be decolorized before use by warming with ignited animal charcoal.

AMMONIUM OXALATE

$(NH_4)_2C_2O_4 + H_2O$. Mol. Wt. 142.16.

Colorless crystals, clearly soluble in 25 parts of cold water. The aqueous solution should be neutral to litmus paper.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium oxalate on ignition should leave no weighable residue.

Sulphates. — Dissolve 5 gm. of ammonium oxalate in 200 cc. of water, heat the solution to boiling, and then add 10 cc. of hydrochloric acid, followed by barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — On adding to a solution of 1 gm. of ammonium oxalate in 25 cc. of water, 10 cc. of nitric acid, and a few drops of silver nitrate solution, no turbidity should develop on shaking.

Heavy Metals. — To a solution of 1 gm. of ammonium oxalate in 25 cc. of water add hydrogen sulphide water; the solution should remain unchanged. Now add to the liquid 5 cc. of ammonia water; no green color should develop, nor should a precipitate form.

Quantitative Determination. — This is to be made as described under Potassium Oxalate Neutral on page 178.

1 cc. of decinormal $KMnO_4 = 0.007108$ gm. of $(NH_4)_2C_2O_4 + H_2O$, log. 85175.

AMMONIUM PHOSPHATE

$(NH_4)_2HPO_4$. Mol. Wt. 132.15.

Colorless crystals, or white, crystalline powder, soluble in 4 parts of cold, or 0.5 part of boiling, water. The aqueous solution is slightly alkaline to litmus paper.

TESTS OF PURITY

Alkalies. — The solution of 2 gm. of ammonium phosphate in 100 cc. of water is treated with lead acetate solution in slight excess to precipitate the phosphoric acid, and is then filtered. The excess of lead in the filtrate is then precipitated with hydrogen sulphide, the liquid filtered, the filtrate evaporated to dryness, and the residue ignited. The residue should not be soluble in water, nor should it have an alkaline reaction.

Arsenic. — On shaking 1 gm. of powdered ammonium phosphate with 3 cc. of stannous chloride solution, the mixture should not darken within one hour.

Carbonates and Sulphates. — On adding hydrochloric acid to a solution of 1 gm. of ammonium phosphate in 20 cc. of water, no effervescence should take place; and on adding barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — On dissolving 1 gm. of ammonium phosphate in 20 cc. of water, and adding 5 cc. of nitric acid, followed by silver nitrate solution, not more than a very slight opalescence should develop.

Nitrates. — Dissolve 2 gm. of ammonium phosphate in 10 cc. of water, and to the solution add 1 drop of indigo solution (1:1000), followed by 10 cc. of concentrated sulphuric acid. The liquid should still retain its blue color on standing one hour.

Heavy Metals. — On dissolving 2 gm. of ammonium phosphate in 20 cc. of water, acidifying the solution with hydrochloric acid, and then adding hydrogen sulphide water, no change should be seen. On now rendering the liquid alkaline with ammonia water, and adding a few drops of ammonium sulphide solution, no green color should develop, nor should a precipitate form.

AMMONIUM SULPHATE

$(NH_4)_2SO_4$. Mol. Wt. 132.14.

Colorless crystals, soluble in 2 parts of cold, and in 1 part of boiling, water; insoluble in alcohol.

TESTS OF PURITY

Non-volatile Matter. — 3 gm. of ammonium sulphate on ignition should leave no weighable residue.

Chlorides. — The solution of 2 gm. of ammonium sulphate in 20 cc. of water should not become turbid on acidifying with nitric acid and then adding silver nitrate solution.

Heavy Metals. — The solution of 2 gm. of ammonium sulphate in 20 cc. of water should remain unaffected by hydrogen sulphide, and by ammonia water with ammonium sulphide solution.

Sulphocyanates. — Dissolve 1 gm. of ammonium sulphate in 10 cc. of water; on adding to the solution several drops of hydrochloric acid and 1 drop of ferric chloric solution, the liquid should not acquire a red color.

Phosphates and Arsenates. — Dissolve 5 gm. of ammonium sulphate in 20 cc. of water, and add to the clear solution 3 cc. of magnesia mixture and 10 cc. of ammonia water. No precipitate should form on standing twelve hours.

Nitrates. — Dissolve 2 gm. of ammonium sulphate in 10 cc. of water, and add to the solution 1 drop of indigo solution (1:1000), followed by 10 cc. of concentrated sulphuric acid. The liquid should still retain its blue color on standing one hour.

AMMONIUM SULPHOCYANATE

(AMMONIUM THIOCYANATE)

NH_4SCN. Mol. Wt. 76.17.

Colorless crystals, easily soluble in water and in alcohol.

TESTS OF PURITY

Non-volatile Matter. 2 gm. of ammonium sulphocyanate, on being ignited, should leave no weighable residue.

Substances insoluble in Alcohol. 1 gm. of ammonium sulphocyanate should completely dissolve in 10 cc. of absolute alcohol, and yield a perfectly clear solution.

Sulphates. On adding a few drops of hydrochloric acid, followed by barium chloride solution, to a solution of 1 gm. of ammonium sulphocyanate in 20 cc. of water, no reaction should be observed within five minutes.

Heavy Metals. On dissolving 1 gm. of ammonium sulphocyanate in 20 cc. of water, and adding to the solution several drops of ammonium sulphide solution, no precipitate should form, nor should a brown color develop.

Iron. The solution of 1 gm. of ammonium sulphocyanate in 20 cc. of water should remain perfectly colorless on adding 0.5 cc. of hydrochloric acid.

AMMONIUM SULPHIDE SOLUTION

(AMMONIUM SULPHYDRATE SOLUTION)

A colorless or yellowish liquid, produced by passing hydrogen sulphide into ammonia water. It is strongly alkaline towards litmus paper.

TESTS OF PURITY

Arsenic, Antimony, and Tin. On adding hydrochloric acid to 50 cc. of ammonium sulphide solution to acidity, hydrogen sulphide gas is liberated in copious amounts, but no colored precipitate should form.

Non-volatile Matter. On evaporating and igniting 10 cc. of ammonium sulphide solution in a porcelain dish, no weighable residue should remain.

Ammonium Carbonate. — On adding 3 cc. of calcium chloride solution to 10 cc. of ammonium sulphide solution, no precipitate should form, even on warming.

AMMONIUM THIOACETATE SOLUTION

(Schiff's Reagent)

$CH_3 \cdot COSNH_4$. Mol. Wt. 93.15.

A clear, yellowish liquid, having a faint odor resembling that of ammonium sulphide, and slightly alkaline to litmus paper. The solution contains about 30 per cent of ammonium thioacetate.

TESTS OF PURITY

Non-volatile Matter. — 10 cc. of ammonium thioacetate solution on evaporation and ignition should leave no weighable residue.

Ammonium Carbonate. — On adding 3 cc. of calcium chloride solution to 10 cc. of ammonium thioacetate solution, no precipitate should form, even on warming.

Sulphates. — A mixture of 10 cc. of ammonium thioacetate solution with 10 cc. of diluted acetic acid should not immediately be rendered turbid on the addition of barium chloride solution.

Note. — The reagent can be kept only a short time unchanged; it readily becomes turbid. It is, therefore, advantageous to prepare only sufficient for eight to ten days' use, which may be done by dissolving the thioacetic acid in a slight excess of ammonia water.

Regarding the use of ammonium thioacetate solution in analysis, see R. Schiff and N. P. Tarugi, Ber., **27**, 3437 (1894) [or Ztschr. anal. Chem., **34**, 456 (1895)]; J. Chem. Soc., **68**, II, 84 (1895). R. Schiff, Ber., **28**, 1204 (1895); J. Chem. Soc., **68**, II, 370 (1895).

ANILINE

$C_6H_5 \cdot NH_2$. Mol. Wt. 93.09.

A colorless, oily, strongly refractive liquid, which rapidly becomes brown on exposure to light and air.

Aniline is soluble in about 35 parts of water. It solidifies in a freezing mixture, and then melts at −8° C. Its specific gravity is 1.027; its boiling point, 183° C.

TESTS OF PURITY

Hydrocarbons and Nitrobenzene. 5 cc. of aniline dissolved in 10 cc. of hydrochloric acid should form a clear fluid which, on being diluted with 15 cc. of water, should not become cloudy on cooling.

NOTE. Regarding the examination of aniline, see Lunge, Chem.-tech. Untersuch.-Meth., 4 ed., **3**, 745 (1900); compare Watts' Dict. of Chem., **1**, 271 (1894). G. Schultz, Chemie des Steinkohlenteers, **1**, 289 (1886); compare Thorpe's Dict. of Appld. Chem., 2 ed., **1**, 162 (1895).

ANTIMONY OXIDE

(ANTIMONOUS OXIDE; ANTIMONY TRIOXIDE)

Sb_2O_3. Mol. Wt. 288.4.

A white powder, insoluble in water, but soluble in hydrochloric acid, tartaric acid, alkali bitartrates, and in solutions of potassium or sodium hydroxide. Antimony trioxide is neutral to litmus paper.

TESTS OF PURITY

Arsenic.—On dissolving 1 gm. of antimony trioxide in 3 cc. of hydrochloric acid (sp. gr. 1.19), and adding 3 cc. of stannous chloride solution, the mixture should not darken within one hour.

Heavy Metals.—Dissolve 1 gm. of antimony trioxide with the aid of heat in 30 cc. of sodium hydroxide solution (sp. gr. 1.3). Dilute the solution with 20 cc. of water and add hydrogen sulphide water. Neither a white nor a brownish-black precipitate should develop.

Chlorides.—Dissolve 1 gm. of antimony trioxide with the aid of heat in 30 cc. of sodium hydroxide solution (sp. gr. 1.3). Add to the solution 70 cc. of nitric acid, filter, and to the filtrate add silver nitrate solution. The liquid may exhibit at most a slight turbidity, but no precipitate should form.

ARSENIC TRIOXIDE

(Arsenous Acid; (Acid) Arsenous Anhydride)

As_2O_3. Mol. Wt. 198.

White, vitreous, or porcelain-like pieces, or white powder, soluble in 15 parts of boiling water.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of arsenic trioxide cautiously heated in a porcelain dish should completely volatilize and leave no weighable residue. (Use a hood!)

Barium Sulphate, Talcum, Calcium Sulphate, etc. — 0.5 gm. of arsenic trioxide should be perfectly soluble in a mixture of 5 cc. of ammonia water and 5 cc. of water, and should yield a clear solution.

Arsenic Sulphide. — Dissolve 5 gm. of arsenic trioxide in a mixture of 5 cc. of sodium hydroxide solution and 15 cc. of water; on adding to the clear solution 2 drops of lead acetate solution, no color reaction should occur.

Quantitative Determination. — Dissolve * 1 gm. of arsenic trioxide with the aid of 6 gm. of potassium bicarbonate in 50 cc. of boiling water, then allow to cool to 15° C., and add sufficient water to make the whole measure 100 cc. Dilute 10 cc. of this solution with 50 cc. of water, and titrate with decinormal iodine solution.

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* It may be quicker to dissolve 1 gm. in a freshly prepared solution of sodium hydroxide (sulphur-free), slightly acidulate with hydrochloric acid, and then add the 6 gm. of bicarbonate. No heat is necessary in this case.

1 cc. of decinormal I   0.00495 gm. of As_2O_3, log. 69461.

Note. — Both the lump and the powder forms of arsenic trioxide must answer the above requirements. The tests here given should be carried out with the powdered preparation, as the whole pieces or lumps dissolve with difficulty.

Regarding the use of arsenic trioxide in volumetric analysis, see Mohr's Lehrb. Chem.-anal. Titriermeth., 7 ed., p. 389 ff. (1896); Sutton, Volumet. Anal., 9 ed., p. 139 (1904).

AZOLITMIN

Azolitmin is a particularly pure, water-soluble coloring matter made from litmus. It occurs in the form of blackish-violet scales.

Azolitmin is used as an indicator, usually in 1 per cent solution. To make this, dissolve 1 gm. of azolitmin in 80 cc. of water with the aid of heat, then add 20 cc. of 85 per cent alcohol, and filter the solution when cold.

TEST OF SENSITIVENESS

Add 0.1 cc. of the above azolitmin solution to 50 cc. of distilled water, as free as possible from alkali and carbon dioxide. The bluish-red color of the liquid should be changed to red by the addition of, at most, 0.05 cc. of decinormal hydrochloric acid, and must be changed to bluish-violet by the addition of, at most, 0.05 cc. of decinormal potassium hydroxide.

Note. The distilled water used in this test must be freed from carbon dioxide by boiling in a platinum vessel, and must be cooled in completely filled flasks with the exclusion of air.

BARIUM ACETATE

$Ba(C_2H_3O_2)_2 + H_2O$. Mol. Wt. 273.46.

A white, crystalline powder, soluble in 2 parts of water and in about 100 parts of alcohol.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of barium acetate in 20 cc. of water, acidulated with nitric acid, must exhibit no turbidity on the addition of silver nitrate solution.

Calcium and Alkalies. — Dissolve 5 gm. of barium acetate in 200 cc. of water, add to the solution 2 cc. of hydrochloric acid and heat to boiling. Now add 15 cc. of 16 per cent sulphuric acid, allow to stand twelve hours, filter, and mix the filtrate with 85 per cent alcohol. At most a faint opalescence should be observed; and on evaporating in a platinum dish and igniting, not more than 0.004 gm. of residue should remain.

Heavy Metals. — 20 cc. of the 1:20 aqueous solution should not acquire a dark color or afford a precipitate on the addition of hydrogen sulphide water; ammonia water with ammonium sulphide solution should produce the same negative results.

Nitrates. — The blue color imparted by 1 drop of 1:1000 indigo solution to a solution of 1 gm. of barium acetate in 10 cc. of water should not disappear on the addition of 10 cc. of concentrated sulphuric acid.

BARIUM CARBONATE

$BaCO_3$. Mol. Wt. 197.4.

A white powder, almost insoluble in water.

TESTS OF PURITY

Solubility in Dilute Hydrochloric Acid. — 5 gm. of barium carbonate should be completely soluble in 10 cc. of hydrochloric acid diluted with 50 cc. of water.

Calcium and Alkalies. — Dissolve 5 gm. of barium carbonate in 10 cc. of hydrochloric acid and 200 cc. of water, and heat the solution to boiling. Then add 15 cc. of 16 per cent sulphuric acid, allow to stand twelve hours, filter, and mix the filtrate with 85 per cent alcohol. Not more than a faint opalescence should be visible, and on evaporating in a platinum dish and igniting, not more than 0.003 gm. of residue should remain.

Heavy Metals. — Neither hydrogen sulphide water, nor ammonia water with ammonium sulphide solution, should produce a dark color or a precipitate on being added to a solution of 1 gm. of barium carbonate in 5 cc. of hydrochloric acid and 15 cc. of water.

Chlorides. — The solution of 1 gm. of barium carbonate in 5 cc. of nitric acid and 15 cc. of water (solution is hastened by heating) should not be affected by silver nitrate solution.

Nitrates. — The blue color imparted by 1 drop of a 1:1000 indigo solution to the solution of 1 gm. of barium carbonate in 10 cc. of diluted acetic acid should not disappear on the addition of 10 cc. of concentrated sulphuric acid.

Note. — Regarding the volumetric determination of barium carbonate, see Mohr's Lehrb. Chem.-anal. Titriermeth., 7 ed., p. 133 (1896); Sutton, Volumet. Anal., 9 ed., p. 70 (1904).

BARIUM CHLORIDE

$BaCl_2 + 2H_2O$. Mol. Wt. 244.33.

Colorless crystals, soluble in 2.5 parts of cold, and in 1.5 parts of hot, water, and insoluble in absolute alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Alkalies. — Dissolve 3 gm. of barium chloride in 100 cc. of water, add 2 cc. of hydrochloric acid and heat to boiling; then add 10 cc. of 16 per cent sulphuric acid, allow to stand twelve hours, and filter. On evaporating the filtrate in a platinum dish and igniting, a residue weighing more than 0.001 gm. should not remain.

Strontium and Calcium Chlorides. — On shaking 1 gm. of powdered barium chloride with 20 cc. of absolute alcohol five minutes, and then filtering, the filtrate on being evaporated and ignited should leave no weighable residue.

Heavy Metals. — 20 cc. of the 1:20 aqueous solution should not afford a dark coloration nor a precipitate when hydrogen sulphide water is added; nor when ammonia water and ammonium sulphide solution are added.

Nitrates. — On adding 1 drop of a 1:1000 indigo solution to a solution of 1 gm. of barium chloride in 10 cc. of water, the blue color must not disappear on the addition of 10 cc. of concentrated sulphuric acid.

Chlorates. — On warming 2 gm. of powdered barium chloride with 10 cc. of concentrated hydrochloric acid in a test tube, neither the crystals nor the liquid should acquire a yellow color; nor should the odor of chlorine become perceptible.

BARIUM DIOXIDE

(Barium Super- or Peroxide)

BaO_2. Mol. Wt. 169.4.

White, or grayish-white powder, insoluble in water, but soluble in cold hydrochloric acid with the formation of hydrogen peroxide. The preparation should contain at least 82 per cent of BaO_2.

TEST OF PURITY

Quantitative Determination. — Introduce 1 gm. of barium peroxide into a graduated flask of 100 cc. capacity, and add to it 5 gm. of potassium iodide; dissolve in 30 cc. of water and 10 cc. of hydrochloric acid, and allow the mixture to stand in the stoppered flask for about half an hour, with frequent shaking. Then fill the flask to the mark, mix, and titrate 10 cc. of the mixture with decinormal sodium thiosulphate solution, using starch solution as the indicator. At least 9.7 cc. of decinormal sodium thiosulphate should be required.

1 cc. of decinormal $Na_2S_2O_3$ = 0.00847 gm. of BaO_2, log. 92788.

BARIUM HYDROXIDE

(Barium Hydrate)

$Ba(OH)_2 + 8H_2O$. Mol. Wt. 315.54.

White crystals, soluble in 20 parts of cold, and in 3 parts of boiling, water, a trace of barium carbonate almost invariably remaining undissolved. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of barium hydroxide in 5 cc. of nitric acid and 15 cc. of water should not be affected by silver nitrate solution.

Calcium and Alkalies. — Dissolve 3 gm. of barium hydroxide in 100 cc. of water, add 5 cc. of hydrochloric acid, and heat to boiling. Then add 10 cc. of diluted sulphuric acid, allow to stand twelve hours, and filter. On evaporating the filtrate in a platinum dish and igniting, a residue weighing more than 0.002 gm. should not remain.

Heavy Metals. — 20 cc. of the 1:20 aqueous solution acidulated with hydrochloric acid should not acquire a dark color, nor yield a precipitate on the addition of hydrogen sulphide water; nor should it do so on adding ammonia water to alkaline reaction, followed by ammonium sulphide solution.

Sulphides. — The 1:20 aqueous solution of barium hydroxide, when acidulated with hydrochloric acid, should not have an odor of hydrogen sulphide, nor should it acquire a dark color on adding lead acetate solution.

Quantitative Determination. — Dissolve 1 gm. of barium hydroxide in 100 cc. of water, add 1 drop of methyl orange solution, and titrate with normal hydrochloric acid. Not less than 6.3 cc. of the normal acid should be necessary to produce the red end-point.

1 cc. of normal HCl = 0.15777 gm. of $Ba(OH)_2 + 8H_2O$, log. 19802.

BARIUM HYDROXIDE SOLUTION

(Baryta Water)

A clear, colorless liquid, of strongly alkaline reaction. Baryta water contains 3.3 per cent of crystallized barium hydroxide, $Ba(OH)_2 + 8H_2O$.

TESTS OF PURITY

The tests to be made are those given under Barium Hydroxide. But for each gram of crystallized barium hydroxide, 30 cc. of baryta water are to be taken. The quantitative determination is made as follows.

Quantitative Determination. — Titrate 50 cc. of baryta water with normal hydrochloric acid, using methyl orange as the indicator. At least 10.5 cc. of normal hydrochloric acid should be required to produce the red end-point.

1 cc. of normal HCl = 0.15777 gm. of $Ba\ (OH)_2 + 8H_2O$, log. 19802.

BARIUM NITRATE

$Ba(NO_3)_2$. Mol. Wt. 261.48.

Colorless crystals, soluble in 20 parts of cold, and in 2.8 parts of boiling, water. The aqueous solution is neutral to litmus paper. Barium nitrate is insoluble in absolute alcohol.

TESTS OF PURITY

Chlorides. — The 1:20 aqueous solution acidulated with nitric acid should show no change on the addition of silver nitrate solution.

Alkalies and Heavy Metals. The test is to be carried out as described under Barium Chloride.

BARIUM SULPHIDE

Gray, hard, amorphous pieces, prepared by fusing a mixture of heavy-spar, powdered coke, and sodium chloride.

On allowing hydrochloric acid to flow over barium sulphide, a uniform current of hydrogen sulphide should be generated.

TEST OF PURITY

Arsenic. Heat 100 cc. of nitric acid (sp. gr. 1.3) in a porcelain dish to 70 to 80° C., and add 10 gm. of finely powdered barium sulphide, gradually, in quantities of 0.2 to 0.3 gm. at a time; when all has been added, heat to boiling. Then add to the mixture 100 cc. of arsenic-free, dilute sulphuric acid (1:5), evaporate on the water-bath as far as possible; continue to heat on the sand-bath until vapors of sulphuric acid begin to be evolved; cool, and finally stir the cooled residue with 100 cc. of water.

Set a Marsh apparatus in operation, using 20 gm. of arsenic-free, granulated zinc and dilute (1:5) sulphuric acid, and then introduce the above residue suspended in water, in small quantities at a time, into the generating flask of the Marsh apparatus. No deposit of arsenic should be observable in the reduction tube after the apparatus has been in operation two hours.

NOTE. This chemical serves for the preparation of arsenic-free hydrogen sulphide. The hydrochloric acid to be used for this purpose is to be tested for arsenic.

BENZENE

(BENZOL)

C_6H_6. Mol. Wt. 78.04.

A clear, colorless liquid, of characteristic odor, insoluble in water, but easily soluble in alcohol and in ether. Benzene solidifies at 0° C. to rhombic, crystalline scales, which melt at + 4° C. Its specific gravity is 0.883, and it boils at 80.5° C.

TESTS OF PURITY

Thiophene.* — On shaking 50 cc. of benzene with 20 cc. of concentrated sulphuric acid, the sulphuric acid should remain colorless; on now adding a crystal of isatin, and again shaking and allowing to stand one hour, the sulphuric acid should have neither a green nor a blue color.

Carbon Disulphide.† — Thoroughly mix 50 cc. of benzene with 50 gm. of alcoholic potassium hydroxide solution (11 gm. of potassium hydroxide in 90 gm. of absolute alcohol), and allow the mixture to stand several hours at a temperature of about 20° C. Shake with about 100 cc. of water, remove the aqueous solution from the benzene, neutralize the latter with acetic acid, and add copper-sulphate solution. No precipitate should form.

BENZIDINE

(PARADIAMINODIPHENYL)

$(C_6H_4)_2 \cdot (NH_2)_2$. Mol. Wt. 184.17.

A grayish-yellow, crystalline powder, melting at 122° C. Benzidine is very difficultly soluble in cold water, but more readily in boiling water, in alcohol, and in ether.

TEST OF PURITY

Sulphur and Sulphates. — Thoroughly mix 5 gm. of benzidine with a mixture of 5 gm. of anhydrous sodium carbonate and 2 gm. of potassium nitrate, and cautiously incinerate in a platinum crucible. When cold, dissolve the melt in 50 cc. of water, filter, add to the filtrate 20 cc. of hydrochloric acid, and heat to boiling. On now adding barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

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* See C. Schwalbe: "On the Sulphur Content of Pure Benzenes"; Ztschr. Farb.-Textl. Ind., **3**, 461 (1904); J. Chem. Soc. **88**, I, 124 (1905). Ztschr. Farb.-Textl. Ind., **4**, 113 (1905); J. Soc. Chem. Ind., **24**, 271 (1905).

† F. Frank: Chem. Ind., **24**, 237, 262 (1901) [or Chem. Centralb., (5) **5**, I, 1251 (1901)]; J. Soc. Chem. Ind., **20**, 566 (1901).

BENZIN

(Petroleum Ether; Petroleum Benzin)

A colorless, non-fluorescent, very inflammable liquid of strong but not unpleasant odor. Specific gravity 0.640 to 0.670. Benzin distils between 40 and 75° C., and does not solidify at 0° C.*

TESTS OF PURITY

Non-volatile Matter and Heavy Oils. — 20 gm. of benzin warmed on the water-bath should volatilize and leave no residue. When dropped upon paper, and allowed to evaporate, it should leave no greasy spot.

Sulphur Compounds or Reducing Agents. — Mix 1 cc. of benzin with 5 cc. of a solution of silver nitrate in alcoholic ammonia; on plunging the test-tube containing the mixture into water of about 50° C., no brown coloration should develop.

BISMUTH SUBNITRATE

(Bismuth Basic Nitrate)

A white, microcrystalline powder, insoluble in water and in alcohol, but soluble in diluted hydrochloric, nitric, or sulphuric acid. On shaking bismuth subnitrate with water, it imparts an acid reaction to the latter.

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* Benzin is a mixture of various hydrocarbons. It has, therefore, no sharp boiling point, but distils within rather wide limits. The testing of benzin for benzene by nitrating the latter with nitro-sulphuric acid was not adopted here, because it is impossible to obtain a benzin that will stand this test.

TESTS OF PURITY

Carbonates, Lead, Copper, Salts of the Alkalies, etc. — 0.5 gm. of bismuth subnitrate should completely dissolve in 25 cc. of cold 16 per cent sulphuric acid without the evolution of carbon dioxide, and should yield a clear solution. 10 cc. of this solution, with an excess of ammonia water, should yield a colorless filtrate. Another 10 cc. of this solution, diluted with 100 cc. of water and treated with hydrogen sulphide to completely precipitate the bismuth, should yield a filtrate leaving no weighable residue on evaporation and ignition.

Chlorides. — On adding silver nitrate solution to a solution of 0.5 gm. of bismuth subnitrate in 5 cc. of nitric acid, not more than a slight opalescent turbidity should develop.

Sulphates. — The solution of 0.5 gm. of bismuth subnitrate in 5 cc. of nitric acid should show no change on the addition of 5 to 10 drops of barium nitrate solution.

Ammonia. — On warming 1 gm. of bismuth subnitrate with 10 cc. of sodium hydroxide solution (sp. gr. 1.3), no ammonia should be evolved (to be ascertained by moistened litmus paper).

Residue on Ignition. — On igniting 1 gm. of bismuth subnitrate, there should remain a residue of bismuth oxide weighing 0.79 to 0.82 gm.

Arsenic. — The residue of bismuth oxide obtained above is triturated to a fine powder, and dissolved in sulphuric acid with heat. Set a Marsh apparatus in operation, using 20 gm. of arsenic-free, granulated zinc, and dilute (1:5) sulphuric acid, then introduce the bismuth solution in small quantities at a time into the evolution flask. No deposit of arsenic should be visible in the reduction tube within half an hour.

BROMINE

Br. Atomic Wt. 79.96.

A dark red, almost black liquid, of specific gravity 2.97 to 2.99, and boiling at 63° C. Bromine dissolves in about 30 parts of water. It is easily soluble in alcohol, ether, chloroform, and carbon disulphide.

TESTS OF PURITY

Non-volatile Matter. — 5 gm. of bromine warmed in a porcelain dish on the water-bath should volatilize and leave no weighable residue.

Sulphuric Acid, Chlorine, and Organic Bromine Compounds (Bromoform; Carbon Tetrabromide). — 5 gm. of bromine dissolve without turbidity of any kind in 100 cc. of water after adding, by drops, 20 cc. of ammonia water.* Evaporate to dryness the solution of ammonium bromide thus obtained, and test for:

(*a*) **Sulphuric Acid.**

Dissolve 2 gm. of the ammonium bromide in 60 cc. of water, and add 0.5 cc. of hydrochloric acid followed by barium chloride solution. A precipitate of barium sulphate should not form on standing twelve hours.

(*b*) **Chlorine.**

Dissolve 0.1 gm. of the ammonium bromide in 10 cc. of water, and mix with 4 cc. of ammonium carbonate solution (1 part of ammonium carbonate, 1 part of ammonia water, and 3 parts of water), then add 12 cc. of decinormal silver nitrate solution, filter, and acidulate the filtrate with nitric acid. Only a slight opalescence should develop.*

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* Organic bromine compounds gradually separate out in the form of oily drops.

Iodine. — Dissolve 1 gm. of bromine in 40 cc. of water, add 4 gm. of powdered iron, and shake two to three minutes. Filter, add starch solution to the filtrate, and cautiously allow a few drops of bromine water to flow upon the surface. A blue zone should not form at once below the yellowish upper portion of the liquid.

BROMINE WATER

A saturated, aqueous solution, containing about 3 per cent of Br.

TESTS OF PURITY

Sulphuric Acid. — Add 0.5 cc. of hydrochloric acid and barium chloride solution to 50 cc. of bromine water, and boil the liquid until the bromine has been completely expelled from it. No precipitate of barium sulphate should separate on standing one hour.

Bromine Content. — Let 10 cc. of bromine water run into a solution of 5 gm. of potassium iodide in 100 cc. of water, allow to stand half an hour in a stoppered flask, and then titrate with decinormal sodium thiosulphate solution.

1 cc. of decinormal $Na_2S_2O_3$ = 0.007996 gm. of Br., log. 90287.

BRUCINE

$C_{23}H_{26}N_2O_4 + 2H_2O$†. Mol. Wt. 430.32.

Small, white crystals, difficultly soluble in cold, but more readily soluble in boiling, water. Brucine is easily soluble in 85 per cent alcohol, and in chloroform. The aqueous solution of brucine is alkaline to litmus paper, and is levogyrate. When dried at 100° C., brucine melts at 178° C.

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* Some opalescence always occurs because silver bromide is not absolutely insoluble in ammonium carbonate solution.

Regarding the quantitative determination of chlorine in bromine, see Topf. Pharm. Ztg., **37**, 364 (1892).

† Brucine also crystallizes with 4 molecules of water; then it dissolves in 320 parts of cold, and in 150 parts of boiling, water.

TESTS OF PURITY

Water of Crystallization. — 1 gm. of brucine dried at 100° C. to constant weight should lose not more than 0.083 gm.

Nitric Acid. — 0.01 gm. of brucine should dissolve in 5 cc. of pure concentrated sulphuric acid, and yield a colorless or, at most, scarcely perceptible faint pink color.*

Strychnine. — Treat 0.5 gm. of brucine with 5 gm. of absolute alcohol at the ordinary temperature for one hour, with frequent shaking. Filter, transfer a portion of the undissolved substance to a watch glass, allow it to dry thereon, and then dissolve it in a few drops of concentrated sulphuric acid. To this solution add a small crystal of potassium dichromate. A play of colors, from blue through violet and red into green, is evidence of the presence of strychnine.

Quantitative Determination. — Dissolve 0.25 gm. of brucine in 50 cc. of 85 per cent alcohol, and titrate with decinormal hydrochloric acid, using lacmoid as the indicator. At least 5.8 cc. of decinormal acid should be required to produce the red end-point.

1 cc. of decinormal HCl = 0.043032 gm. of $C_{23}H_{26}N_2O_4 + 2H_2O$, log. 63379.

CADMIUM BOROTUNGSTATE SOLUTION

A perfectly clear, yellowish, or light brown, liquid, having a specific gravity of 3.28.

CADMIUM AND POTASSIUM IODIDE

(Potassium–Cadmium Iodide)

A white powder, easily soluble in water and in alcohol.

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* The sulphuric acid should be tested with diphenylamine to insure the absence of nitric acid.

Cadmium and potassium iodide readily acquires a slight yellowish color on keeping.

TESTS OF PURITY

Foreign Metals. —

(*a*) Dissolve 1 gm. of cadmium and potassium iodide in 20 cc. of water, add to the solution 2 cc. of potassium hydroxide solution, and filter. Neither before nor after acidulating with hydrochloric acid should the filtrate yield a precipitate with hydrogen sulphide water.

(*b*) Dissolve 1 gm. of cadmium and potassium iodide in 30 cc. of water, and add to the solution 5 cc. of ammonia water. The liquid should remain clear and colorless on shaking.

Sulphates. — The 1:20 aqueous solution acidulated with hydrochloric acid should not be affected by barium chloride solution.

Iodic Acid. — The 1:20 solution freshly prepared with boiled water should not immediately give a blue color on adding starch solution followed by 2 or 3 drops of dilute sulphuric acid.

CALCIUM CARBONATE PRECIPITATED

$CaCO_3$. Mol. Wt. 100.1.

A white, crystalline powder, almost insoluble in pure water (1:27000), but somewhat soluble in carbonated water.

TESTS OF PURITY

Solubility in Hydrochloric, Nitric, and Acetic Acids. — 5 gm. of calcium carbonate should be completely dissolved by 25 cc. of hydrochloric acid, and by 25 cc. of nitric acid, and by 60 cc. of 30 per cent acetic acid. The solutions must be clear and colorless.

Heavy Metals. — Dissolve 1 gm. of calcium carbonate in 5 cc. of hydrochloric acid and 25 cc. of water. This solution should not afford a precipitate or a green color with hydrogen sulphide water; nor with ammonia water in excess accompanied by 2 to 3 drops of ammonium sulphide solution.

Magnesium. — Dissolve 1 gm. of calcium carbonate in 5 cc. of hydrochloric acid and 5 cc. of water. To this solution add 10 cc. of ammonia water and an excess of ammonium oxalate solution, allow to stand five hours, filter, and add to the filtrate sodium phosphate solution. No precipitate should form on standing twelve hours.

Sulphates. — Dissolve 1 gm. of calcium carbonate in 5 cc. of hydrochloric acid and 25 cc. of water, boil the solution five minutes, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — 1 gm. of calcium carbonate dissolved in 5 cc. of nitric acid and 25 cc. of water, must show no change on the addition of silver nitrate solution.

Phosphates. — Dissolve 10 gm. of calcium carbonate in 50 cc. of nitric acid, add 25 cc. of ammonium molybdate solution, and allow to stand twelve hours at a temperature of 30 to 40° C. No yellow precipitate should form.

Alkalies and Calcium Oxide. — 1 gm. of calcium carbonate shaken with 50 parts of boiled and cooled water should yield a filtrate without an alkaline reaction, which on evaporation and ignition should leave a residue weighing at most 0.001 gm.

CALCIUM CHLORIDE, CRYSTALS

$CaCl_2 + 6\,H_2O$. Mol. Wt. 219.09.

Colorless crystals, deliquescent in the air, and easily soluble in water and in alcohol. The 1 : 10 solution should be neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Absolute Alcohol. — 2 gm. of calcium chloride should completely dissolve in 20 cc. of absolute alcohol.

Heavy Metals. — 20 cc. of the 1:10 aqueous solution should show no change on adding 1 cc. of hydrochloric acid and hydrogen sulphide water. On further adding 5 cc. of ammonia water and 2 to 3 drops of ammonium sulphide solution, no green color should develop, nor should a precipitate form.

Sulphates. — On adding barium chloride solution to the solution of 2 gm. of calcium chloride in 20 cc. of water acidulated with 0.5 cc. of hydrochloric acid, no precipitate should form on standing twelve hours.

Ammonium Salts. — On boiling 2 gm. of calcium chloride with 10 cc. of sodium hydroxide solution, no ammonia should be evolved (to be ascertained by moistened litmus paper).

Barium. — On adding 20 cc. of calcium sulphate solution to the solution of 2 gm. of calcium chloride in 20 cc. of water, no precipitate should form within three hours.

Arsenic. — Introduce 10 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen with dilute (1:5) sulphuric acid. Dissolve 5 gm. of calcium chloride in 20 cc. of water, introduce the solution in small quantities at a time into the Marsh apparatus, and maintain the stream of gas for about one hour. No deposit of arsenic should be visible in the reduction tube within this time.

CALCIUM CHLORIDE, DRY, GRANULATED

White, granular, porous masses in pieces the size of peas, the form preferred for drying gases. This is the preparation to be used for filling absorption tubes for the determination of carbon dioxide, as in elementary analyses, but it should be previously rendered neutral by means of carbonic acid.

CALCIUM CHLORIDE FUSED

White crystalline masses; formula: practically $CaCl_2$. Fused calcium chloride is chiefly used for drying liquids, *e.g.*, ethers, esters, ethereal oils, hydrocarbons, etc.

CALCIUM HYDROXIDE

(Slaked Lime)

$Ca(OH)_2$. Mol. Wt. 74.11.

A white, dry powder.

The article is tested as to purity by the methods detailed under Calcium Oxide from Marble, on page 83.

CALCIUM OXIDE, FROM ICELAND SPAR

CaO. Mol. Wt. 56.1.

White pieces in the form of Iceland spar crystals.

TESTS OF PURITY

Solubility and Sulphates. — 3 gm. of calcium oxide slaked with 10 cc. of water should completely dissolve in 15 cc. of hydrochloric acid without effervescence. Dilute the solution with 50 cc. of water, heat to boiling, and add barium chloride solution. On standing twelve hours, not more than a scarcely perceptible, unweighable trace of barium sulphate should be present.

Phosphates. — Slake 3 gm. of calcium oxide with 10 cc. of water, dissolve in 25 cc. of nitric acid, and add 25 cc. of ammonium molybdate solution. No yellow precipitate should form on standing twelve hours at 30 to 40° C.

Chlorides. — Slake 3 gm. of calcium oxide with 10 cc. of water, dissolve in 20 cc. of nitric acid, and dilute the solution with 10 cc. of water. The solution should show no change on the addition of silver nitrate solution.

Iron. — Slake 1 gm. of calcium oxide with 5 cc. of water, and dissolve in 10 cc. of hydrochloric acid. The solution should not immediately afford a blue color on adding 0.5 cc. of potassium ferrocyanide solution.

CALCIUM OXIDE, FROM MARBLE

(LIME)

CaO. Mol. Wt. 56.1.

White pieces of granular structure which develop much heat on being sprinkled with water, and which become converted into a fine, dust-like powder, or a pasty mass, according to the quantity of water used. Calcium oxide is soluble in about 800 parts of cold, and in 1300 parts of boiling, water.

TESTS OF PURITY

Carbonates, Silica, Alumina, and Sulphates. — Slake 5 gm. of calcium oxide with 10 cc. of water. The magma so obtained should almost completely dissolve in 30 cc. of hydrochloric acid without strong effervescence. Dilute the solution with 20 cc. of water, and filter. (The insoluble residue, after ignition, should weigh at most 0.005 gm.) To 25 cc. of the filtrate, add barium chloride solution; the liquid should show no change within ten minutes. Supersaturate 25 cc. of the filtrate with ammonia water; the liquid should not be rendered more than slightly opalescent.

Chlorides. — Slake 1 gm. of calcium oxide with 3 cc. of water, dissolve in 10 cc. of nitric acid, dilute the solution with 10 cc. of water, filter, and add silver nitrate solution to the filtrate. The liquid should not be rendered more than slightly opalescent.

CALCIUM PHOSPHATE, DIBASIC

(SECONDARY CALCIUM PHOSPHATE; DICALCIUM PHOSPHATE)

$CaHPO_4 + 2H_2O$. Mol. Wt. 172.14.

A white, crystalline powder, easily soluble in hydrochloric, nitric, or phosphoric acid, without effervescence; it is difficultly soluble in water, and in cold acetic acid.

Dicalcium phosphate does not dissolve in boiling water without decomposition; it produces a liquid of acid reaction and an amorphous residue, the composition of which approximates that of the neutral salt.

TESTS OF PURITY

Arsenic. — The test is carried out as described under Calcium Phosphate, Monobasic on page 85, using a solution of 2 gm. of dicalcium phosphate in 5 cc. of hydrochloric acid and 15 cc. of water

Chlorides. — Dissolve 1 gm. of dicalcium phosphate in 5 cc. of nitric acid and 15 cc. of water; on adding silver nitrate solution, not more than a slight opalescent turbidity should be visible.

Heavy Metals. — The solution of 1 gm. of dicalcium phosphate in 5 cc. of hydrochloric acid and 15 cc. of water should afford a pure white precipitate on the addition of hydrogen sulphide water and 10 cc. of ammonia water.

Sulphates. — On shaking 1 gm. of dicalcium phosphate with 20 cc. of water and filtering, the filtrate, on adding 1 cc. of hydrochloric acid and barium chloride solution, should develop no precipitate within twelve hours.

Residue on Ignition. — On ignition, dicalcium phosphate should yield 74 to 75 per cent of its weight of residue.*

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* On ignition, dicalcium phosphate is converted into calcium pyrophosphate, whereby it theoretically loses 26.12 per cent of water.

CALCIUM PHOSPHATE, MONOBASIC

(Calcium Biphosphate; Calcium Acid (or Super) Phosphate; Primary Calcium Phosphate; Monocalcium Phosphate)

$Ca(H_2PO_4)_2 + H_2O$. Mol. Wt. 252.14.

Colorless, pearly scales, which readily deliquesce in the air.

Calcium biphosphate dissolves in much water, apparently without decomposition; with a small quantity of water, an amorphous salt separates, richer in calcium, while the solution exhibits a strongly acid reaction.

On heating a 1:20 aqueous solution of calcium biphosphate to boiling, a precipitate forms.

TESTS OF PURITY

Arsenic.—Introduce 10 gm. of arsenic-free, granulated zinc into the generating flask of a Marsh apparatus, and start the stream of hydrogen by adding dilute (1:5) sulphuric acid. After the apparatus and reagents have been tested in the usual manner, dissolve 2 gm. of calcium biphosphate in 5 cc. of hydrochloric acid and 15 cc. of water. Introduce this solution into the Marsh apparatus, and maintain the flow of gas for about one hour. No deposit of arsenic should be visible in the reduction tube within this time.

Chlorides. — A solution of 1 gm. of calcium biphosphate in 5 cc. of nitric acid and 15 cc. of water should not be rendered more than slightly opalescent on the addition of silver nitrate solution.

Heavy Metals. — On adding hydrogen sulphide water and 10 cc. of ammonia water to a solution of 1 gm. of calcium biphosphate in 5 cc. of hydrochloric acid and 15 cc. of water, a pure white precipitate should form.

Note. — The presence of a small quantity of sulphuric acid in this preparation cannot be avoided. It is due to the customary methods of manufacturing the salt.

CALCIUM PHOSPHATE, TRIBASIC

(TERTIARY CALCIUM PHOSPHATE; TRICALCIUM PHOSPHATE)

$Ca_3(PO_4)_2$. Mol. Wt. 310.3.

A white, amorphous powder, insoluble in cold water. Tricalcium phosphate is gradually decomposed by boiling water into an insoluble basic salt and an easily soluble acid salt. It is easily soluble in hydrochloric or nitric acid, and without effervescence.

TESTS OF PURITY

Arsenic. — The test is carried out as detailed under Calcium Phosphate, Monobasic, on page 85, using a solution of 2 gm. of tricalcium phosphate in 5 cc. of hydrochloric acid and 15 cc. of water.

Sulphates. — Shake 1 gm. of tricalcium phosphate with 20 cc. of water, filter, and add to the filtrate 1 cc. of hydrochloric acid and barium chloride solution. No precipitate should form on standing twelve hours.

Chlorides. — The solution of 1 gm. of tricalcium phosphate in 5 cc. of nitric acid and 15 cc. of water should not show more than a slight opalescent turbidity on adding silver nitrate solution.

Heavy Metals. — The solution of 1 gm. of tricalcium phosphate in 5 cc. of hydrochloric acid and 15 cc. of water should yield a pure white precipitate on adding hydrogen sulphide water and 10 cc. of ammonia water.

CALCIUM SULPHATE

(GYPSUM)

$CaSO_4 + 2H_2O$. Mol. Wt. 172.19.

A fine, white powder, difficultly soluble in water (about 1:500).

TESTS OF PURITY

Iron, Magnesium, and Alkalies. — On warming 2 gm. of calcium sulphate with 10 cc. of hydrochloric acid and 100 cc. of water, a clear solution should result, which, on the addition of 15 cc. of ammonia water and a few drops of ammonium sulphide solution, should not acquire a greenish or dark color. To the solution, no matter whether a precipitate of calcium sulphate has formed or not, add ammonium oxalate solution in slight excess, filter, evaporate the filtrate, and ignite in a platinum dish. There should not remain a residue weighing more than 0.001 gm.

CALCIUM SULPHIDE

(Sulphurated Lime)

Light-gray cubes, from which dilute hydrochloric acid sets free copious quantities of hydrogen sulphide gas.

TEST OF PURITY

Arsenic. — This is carried out as detailed under Barium Sulphide on page 72.

Note. — This preparation serves for the generation of arsenic-free hydrogen sulphide. The hydrochloric acid used for this purpose should also be tested for arsenic.

CARBON DISULPHIDE

CS_2. Mol. Wt. 76.12.

A clear, colorless, neutral liquid of specific gravity 1.270 to 1.272, and boiling at 46 to 47° C.

TESTS OF PURITY

Non-volatile Matter. — On evaporating 50 cc. of carbon disulphide on the water-bath, no weighable residue should remain.*

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* Under the influence of light, carbon disulphide becomes yellowish, and then leaves a slight residue on evaporation.

Hydrogen Sulphide and Foreign Organic Sulphur Compounds. —

(*a*) On shaking 10 cc. of carbon disulphide with lead carbonate, the latter should not acquire a brown color.

(*b*) On shaking 2 cc. of carbon disulphide in a dry vessel with a globule of metallic mercury, the bright surface of the latter should not become covered with a dark, pulverulent coating.

Sulphuric and Sulphurous Acids. On shaking 10 cc. of carbon disulphide with 5 cc. of water, the latter should neither redden nor decolorize blue litmus paper.

CARMINE

(CARMINE I. [NACCARAT])

Bright red, light pieces, which are very friable and reducible to a fine powder. Carmine is insoluble in water, and in dilute acids, but is soluble in ammonia.

TESTS OF PURITY

Solubility. — 0.15 gm. should be almost completely soluble in a mixture of 5 cc. of ammonia water and 20 cc. of water, yielding a violet-red solution; only a small amount of flocculent matter should remain undissolved.

Ash. — 0.25 gm. of carmine cautiously incinerated in a porcelain crucible should not yield an ash weighing more than 0.02 gm. Particular attention should be paid to the odor of the decomposing carmine; it is similar to that yielded during the combustion of proteids. An odor of bromine would point to a sophistication with an eosine-lake; and an odor of phenol would point to an admixture of peonine-lake.

NOTE. — Regarding the adulterations of cochineal-carmine, see E. Donath, Dingl. pol. J., **294**, 188 (1894); J. Soc. Chem. Ind., **14**, 305 (1895).

Regarding the preparation of carmine solutions and carmine tinctures, see Merck's Reagentien-Verzeichnis, p. 171 (1903); Cohn, Tests and Reagents, p. 349 (1903).

Regarding the composition of carmine and of carmine ash, see S. Feitler, Ztschr. angew. Chem., **5**, 136 (1892) [or Ztschr. anal. Chem., **32**, 627 (1893)]; J. Soc. Chem. Ind., **12**, 256 (1893).

CHARCOAL, ANIMAL

(Blood Charcoal, Purified by Acid)

A dry, light, black powder.

TESTS OF PURITY

Material Soluble in Water. — Exhaust 1 gm. of animal charcoal by boiling with 20 cc. of water, filter, and evaporate the filtrate to dryness. The residue should not weigh more than 0.003 gm.

Material Soluble in Alcohol. — Heat a mixture of 1 gm. of animal charcoal and 20 cc. of alcohol to boiling, and filter. The filtrate should leave no weighable residue on evaporation.

Sulphates, Chlorides, and Nitrates. —

(*a*) Boil 1 gm. of animal charcoal with 50 cc. of water for a few minutes, and filter. The filtrate should be colorless and neutral. On adding to 10 cc. of the filtrate some barium nitrate solution, no immediate turbidity should be produced.

(*b*) On adding silver nitrate solution to 10 cc. of the filtrate, not more than a slight opalescence should develop.

(*c*) On adding 1 drop of 1:1000 indigo solution and 5 cc. of concentrated sulphuric acid to 10 cc. of the filtrate, the blue color of the solution should not disappear.

Copper, Iron, and Calcium. — Boil 1 gm. of animal charcoal with 40 cc. of water and 10 cc. of hydrochloric acid for about five minutes, filter, and to 10 cc. of the filtrate add 25 cc. of ammonia water; the liquid should not acquire a blue color, nor should a flocculent precipitate form. On the further addition of a few drops of ammonium sulphide and ammonium oxalate solutions, no turbidity should develop immediately.

Residue on Ignition. — 1 gm. of animal charcoal should leave a residue weighing not more than 0.10 gm. on ignition.

Hydrogen Sulphide. — Heat 1 gm. of animal charcoal with 40 cc. of water and 10 cc. of hydrochloric acid, and test the escaping vapors with moistened lead acetate paper. The latter should not acquire a brown color.

Decolorizing Power. — Dissolve 50 gm. of caramel (sugar coloring; burnt sugar) in 50 cc. of water, add 100 cc. of 85 per cent alcohol, dilute the whole to 1 liter, allow to stand for several days, and then filter. Dilute 5 cc. of this caramel solution with 50 cc. of water, add 1 gm. of animal charcoal, boil the mixture ten minutes under a reflux condenser, and then filter. The filtrate should be perfectly colorless.

CHLORINE WATER

A clear, pale, greenish-yellow liquid, having a strong odor of chlorine. The liquid should contain about 0.4 per cent of Cl.

TESTS OF PURITY

Non-volatile Matter. — 20 gm. of chlorine water evaporated in a glass dish on a water-bath should leave no weighable residue.

Chlorine Content. — Let 25 gm. of chlorine water run into a flask containing a solution of 1 gm. of potassium iodide in 25 cc. of water. Add a few drops of starch solution, and titrate the liberated iodine with decinormal sodium thiosulphate solution, of which at least 28.2 cc. should be used to cause the blue color to disappear.

1 cc. of decinormal $Na_2S_2O_3$ = 0.003545 gm. of Cl, log. 54962.

Hydrochloric Acid.* — Shake 20 gm. of chlorine water with about 5 gm. of pure mercury vigorously for five minutes. Filter, add phenolphthalein to the solution, and run in normal potassium hydroxide solution, by drops, until a red color appears. Not more than 0.1 cc. of normal alkali should be required to produce the red end-point.

CHLOROFORM

$CHCl_3$. Mol. Wt. 119.35.

A clear, colorless, very volatile liquid, slightly soluble in water (1:200), but miscible in all proportions with alcohol, ether, and fatty and volatile oils. Chloroform has a specific gravity of 1.485 to 1.489, and boils at 60 to 62° C.†

TESTS OF PURITY

Free Acid. — Vigorously shake together 20 cc. of chloroform and 10 cc. of water for half a minute; after the chloroform has separated, draw off the aqueous layer with a pipette. The liquid should not redden blue litmus paper, nor should it acquire an opalescence when overlaid on a mixture of 2.5 cc. of water and 2.5 cc. of silver nitrate solution.

Free Chlorine. — On shaking 5 cc. of chloroform with 5 cc. of zinc iodide-starch solution, the latter should not acquire a blue color, nor should the chloroform become colored.

Foreign Organic Bodies. — On repeatedly shaking 20 cc.

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* Chlorine water, unless recently prepared, always contains hydrochloric acid.

† The readiness of pure (absolute) chloroform to decompose may be prevented by the addition of a small amount (up to 1 per cent) of pure absolute alcohol. The specific gravity and boiling point are somewhat changed by this addition. The alcohol may be removed from the chloroform by shaking it twice with double its volume of pure sulphuric acid, neutralizing the acid with granulated potassium carbonate, and distilling.

of chloroform and 15 cc. of concentrated sulphuric acid in a glass-stoppered flask, 3 cm. wide, which has been rinsed previously with sulphuric acid, the acid should not acquire a color within one hour.

CHROMIUM TRIOXIDE — FREE FROM SULPHURIC ACID

((ACID) CHROMIC ANHYDRIDE)

CrO_3. Mol. Wt. 100.01.

Dark, brownish-red needles, or rhombic prisms, very readily soluble in water. Chromium trioxide contains almost 100 per cent of CrO_3.

TESTS OF PURITY

Sulphuric Acid. — 2 gm. of chromium trioxide must afford a clear solution with 20 cc. of water. On adding to the solution 20 cc. of hydrochloric acid and 1 cc. of barium chloride solution, no turbidity should occur immediately.

Potassium Sulphate and Potassium Chromate. — Ignite 0.2 gm. of chromium trioxide in a porcelain crucible, triturate the residue with about 20 cc. of water, and filter. Evaporate the filtrate to dryness on a water-bath, dry the residue at 100° C., and weigh. The residue must not weigh more than 0.002 gm.

Quantitative Determination. — Dissolve 5 gm. of chromium trioxide in water and dilute to 500 cc. Introduce 10 cc. of this solution into a stoppered flask of about 400 cc. capacity, dilute with 100 cc. of water, and add 5 gm. of potassium iodide and 2 cc. of hydrochloric acid. Allow the mixture to stand for about ten minutes with frequent shaking, then dilute with 200 cc. of water, and titrate with decinormal sodium thiosulphate solution.

1 cc. of decinormal $Na_2S_2O_3$ = 0.003336 gm. of CrO_3, log. 52323.

COBALT NITRATE

(COBALTOUS NITRATE)

$Co(NO_3)_2 + 6H_2O$. Mol. Wt. 291.17.

Red, monoclinic prisms, deliquescent in moist air, and readily soluble in water and in alcohol.

TESTS OF PURITY

Sulphates. — A solution of 1 gm. of cobalt nitrate in 20 cc. of water, to which have been added 0.5 cc. of hydrochloric acid and a little barium chloride solution, should not become turbid.

Alkali Salts. — Precipitate the cobalt completely from a solution of 2 gm. of cobalt nitrate in 100 cc. of water by adding ammonia water and ammonium sulphide solution; filter, evaporate the filtrate to dryness, and ignite the residue. The weight of the latter should not exceed 0.005 gm.

Zinc. — To a solution of 0.5 gm. of cobalt nitrate in 50 cc. of water, add 5 cc. of sodium hydroxide solution (sp. gr. 1.3); filter, and to the filtrate add ammonium sulphide solution. No precipitate should form.

Lead and Copper. — Dissolve 2 gm. of cobalt nitrate in 50 cc. of water, and add 2 cc. of nitric acid, followed by hydrogen sulphide water. The solution must show no change.

Nickel.* — Dissolve 1 gm. of cobalt nitrate in 20 cc. of water, add 3 gm. of potassium cyanide, boil the solution until it has acquired a yellow color, filter, and to the filtrate add potassium hydroxide solution and bromine water. No brown color should develop.

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* Compare the method using nitrosobeta-naphthol. See Prescott and Johnson, Qualitative Chemical Analysis, p. 166 (1901).

COLLODION

A colorless, or slightly yellowish, neutral, syrupy liquid, which, exposed in thin layers, evaporates, and leaves a tough, colorless film. Collodion contains about 4 per cent of soluble cotton.

TESTS OF PURITY

Acids. — Blue litmus paper, when immersed in collodion, should not be reddened immediately.

Determination of Residue. — On evaporating 10 gm. of collodion on the water-bath, it should leave a residue, which, when dried at 100° C., should weigh 0.38 to 0.40 gm.

COPPER, BY ELECTROLYSIS

Cu. Atomic Wt. 63.6.

TESTS OF PURITY

Foreign Metals.

(*a*) Dissolve 10 gm. of copper in 60 cc. of nitric acid (sp. gr. 1.3), and evaporate the solution to dryness on the water-bath. The residue should completely dissolve in 50 cc. of water and 10 cc. of nitric acid (sp. gr. 1.3), yielding a clear solution (absence of antimony and tin). To the solution add 15 cc. of concentrated sulphuric acid (sp. gr. 1.84), evaporate on the water-bath as far as possible, heat the residue on a sand-bath until vapors of sulphuric acid begin to be evolved, and take up the residue with 100 cc. of water. No insoluble residue should remain (absence of lead). On adding 5 cc. of hydrochloric acid to the clear solution, no turbidity should ensue (absence of silver). Now add 150 cc. of ammonia water to the liquid, allow to stand three to four hours at 50 to 60° C., filter through an ashless filter, wash with ammoniacal water until perfectly free from copper. Incinerate the filter with any precipitate it may contain, and ignite. The residue should not weigh more than 0.001 gm. (iron and bismuth).

(*b*) Dissolve 10 gm. of copper in 60 cc. of nitric acid (sp. gr. 1.3), add to the solution 15 cc. of concentrated sulphuric acid (sp. gr. 1.84), evaporate the solution on the water-bath as far as possible, and heat the residue on a sand-bath until the vapors of sulphuric acid begin to be evolved. Dissolve the residue in 300 cc. of water, pass hydrogen sulphide gas into the solution at 70° C. until the copper is completely precipitated, filter, concentrate the filtrate, expel the sulphuric acid on the sand-bath, and ignite the residue. The weight of the latter should not exceed 0.002 gm.

Arsenic. Dissolve 10 gm. of copper in 60 cc. of nitric acid (sp. gr. 1.3), add to the solution 15 cc. of concentrated sulphuric acid (sp. gr. 1.84), evaporate the solution on the water-bath as far as possible, and then heat the residue on the sand-bath until vapors of sulphuric acid are evolved. When cold, dissolve the residue in 100 cc. of water.

Set a Marsh apparatus in operation, using 50 gm. of arsenic-free, granulated zinc, and dilute (1:5) sulphuric acid, then introduce the copper sulphate solution in small quantities at a time, and maintain the flow of gas for about two hours. No deposit of arsenic should be visible in the reduction tube within this time.

COPPER CHLORIDE, CUPRIC

(Copper Dichloride)

$CuCl_2 + 2H_2O$. Mol. Wt. 170.53.

Green, hygroscopic crystals, easily soluble in water, alcohol, and ether.

TESTS OF PURITY

Substances Insoluble in Alcohol. — The solution of 5 gm. of copper chloride in 5 cc. of water should not be rendered turbid on mixing with 5 cc. of 95 per cent alcohol.

Sulphates. — Dissolve 1 gm. of copper chloride in 20 cc. of water, and add 0.5 cc. of hydrochloric acid, followed by barium chloride solution. No turbidity should ensue within five minutes.

Salts of Alkali Metals. — Dissolve 3 gm. of copper chloride in 100 cc. of water, add 5 cc. of hydrochloric acid, and into the solution, maintained at about 70° C., pass hydrogen sulphide gas, until the copper is completely precipitated; filter, evaporate the filtrate to dryness, and ignite the residue. The weight of the latter should not exceed 0.002 gm.

Iron. — The test is carried out as described under Copper and Ammonium Chloride.

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen by adding dilute (1:5) sulphuric acid. Dissolve 1 gm. of copper chloride in 20 cc. of water, introduce the solution in small quantities at a time into the Marsh apparatus, and maintain a slow stream of gas for about one hour. No deposit of arsenic should be visible in the reduction tube of the apparatus within this time.

COPPER CHLORIDE, CUPROUS

(COPPER MONOCHLORIDE)

Cu_2Cl_2. Mol. Wt. 198.1.

A white, crystalline powder, insoluble in water, but soluble in concentrated hydrochloric acid and in ammonia water.

The quality of cuprous chloride may be judged from its appearance. The preparation should be white, but not green nor brown.* Both the hydrochloric acid and the ammoniacal solutions should rapidly and freely absorb carbon monoxide.

COPPER OXIDE

(CUPRIC OXIDE)

CuO. Mol. Wt. 79.6.

Copper oxide is used in ultimate organic analysis, in the form of fine powder or coarse granules, and also in the form of wire.

TESTS OF PURITY

Nitrates, Chlorides, and Carbon Dioxide. — On heating 100 gm. of copper oxide, and passing over it a stream of moist air freed from carbonic acid, no vapors should be given off which redden litmus paper, or render lime water turbid.

Substances not Precipitated by Hydrogen Sulphide (Iron, etc.). — Dissolve 2 gm. of copper oxide in 10 cc. of hydrochloric acid (sp. gr. 1.19), dilute with water to 100 cc.; ignite the insoluble residue, the weight of which should not exceed 0.005 gm. Pass hydrogen sulphide gas into the solution at about 70° C., until the copper has completely precipitated, filter, evaporate the filtrate on the water-bath, and ignite the residue. The weight of the latter should not exceed 0.02 gm.

Sulphates. — Dissolve 1 gm. of copper oxide in 5 cc. of hydrochloric acid (sp. gr. 1.19), dilute with water to 50 cc., and add barium chloride solution. No immediate turbidity should appear.

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* Cuprous chloride soon acquires a green color in air.

Calcium. — Digest 20 gm. of copper oxide with a mixture of 5 cc. of nitric acid and 95 cc. of water for about fifteen minutes, shaking frequently; filter, precipitate the copper in the filtrate completely by passing hydrogen sulphide gas, and filter again. Evaporate the filtrate on the water-bath to about 20 cc., add ammonia water in excess, filter once more, and to the filtrate add ammonium oxalate solution. No immediate turbidity should be produced.

COPPER SULPHATE

(Cupric Sulphate)

$CuSO_4 + 5H_2O$. Mol. Wt. 249.74.

Blue, transparent crystals, slightly efflorescent in dry air, soluble in 3.5 parts of cold, and in 1 part of boiling, water; insoluble in alcohol. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Salts of the Alkalis, Earths, etc. — Dissolve 3 gm. of copper sulphate in 100 cc. of water, add 5 cc. of hydrochloric acid, and into the solution, maintained at about 70° C., pass hydrogen sulphide gas until the copper is completely precipitated. On evaporating the filtrate to dryness, and igniting the residue, the weight of the latter should not exceed 0.001 gm.

Iron. — To the solution of 5 gm. of copper sulphate in 25 cc. of water, add 2 cc. of nitric acid (sp. gr. 1.3), and heat the mixture to boiling. Then add 20 cc. of ammonia water, pour the liquid through an ashless filter, wash the latter with ammoniacal water until perfectly free from copper, and then incinerate and ignite the filter together with any precipitate it may contain. The weight of the residue should not exceed 0.001 gm.

COPPER AND AMMONIUM CHLORIDE

(Ammonio-Cupric Chloride)

$CuCl_2 \cdot 2(NH_4Cl) + 2H_2O$. Mol. Wt. 277.57.

Blue crystals, giving with water a clear solution slightly acid to litmus paper.

TESTS OF PURITY

Free Acids. — A solution of 30 gm. of copper and ammonium chloride in 100 cc. of water must be perfectly clear. On introducing into the solution several pieces of piano wire, the latter should dissolve with the deposition of copper, but without any evolution of gas.

Sulphates. — Dissolve 1 gm. of copper and ammonium chloride in 20 cc. of water, and add 0.5 cc. of hydrochloric acid, followed by barium chloride solution. No turbidity should be produced within five minutes.

Salts of the Alkalis, Earths, etc. — Dissolve 3 gm. of copper and ammonium chloride in about 100 cc. of water, add 5 cc. of hydrochloric acid, pass hydrogen sulphide gas into the solution at about 70° C., until the copper is completely precipitated; filter, evaporate the filtrate, and ignite the residue. The weight of the latter should not exceed 0.002 gm.

Iron. — Dissolve 5 gm. of copper and ammonium chloride in 25 cc. of water, add 2 cc. of nitric acid (sp. gr. 1.3), heat to boiling, then add 20 cc. of ammonia water and pour the liquid through an ashless filter. Wash the latter with ammoniacal water until perfectly free from copper; incinerate and ignite the filter together with any precipitate it may contain. The weight of the residue should not exceed 0.002 gm.

DIPHENYLAMINE *

$(C_6H_5)_2NH$. Mol. Wt. 169.12.

White, monoclinic crystals, insoluble in water, but easily soluble in alcohol, ether, and benzene. Diphenylamine melts at 54° C. and boils at 302° C.

TESTS OF PURITY

Nitric Acid. — 0.2 gm. of diphenylamine should dissolve in 20 cc. of pure concentrated sulphuric acid with 2 cc. of water, and yield a colorless solution. Should a blue color develop, the sulphuric acid should be tested with brucine for the possible presence of nitric acid.

Aniline. — On pouring 1 gm. of powdered diphenylamine into 20 cc. of a chlorinated lime solution, the liquid should not acquire a violet color.

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* The diphenylamine solution, used as a reagent, is prepared by dissolving 0.5 gm. of diphenylamine in 100 cc. of pure, concentrated sulphuric acid (free from nitric acid) and 20 cc. of water. According to G. Lunge [Ztschr. angew. Chem., **7**, 345 (1894); J. Chem. Soc., **66**, II, 398 (1894)], the reagent is employed as follows:

To test for nitric or nitrous acid, introduce about 5 cc. of the reagent solution into a test tube, and overlay it with the liquid to be examined. If the latter is specifically heavier than the reagent solution, the diphenylamine solution is to be overlaid upon the liquid.

Regarding the increased sensitiveness of the nitric-acid reaction with diphenylamine in water analyses, see R. Cimmino, Ztschr. anal. Chem., **38**, 429 (1899); J. Chem. Soc., **70**, II, 805 (1899).

ETHER

(ETHYL ETHER)

$(C_2H_5)_2O$. Mol. Wt. 74.08.

I

ETHER, SP. GR. 0.720

A clear, colorless, mobile liquid of specific gravity 0.720,* boiling at 34 to 36° C. Ether should not redden moist blue litmus paper. Filter paper which has been moistened with ether should have no odor when dry.

TESTS OF PURITY

Residue. — On allowing 20 cc. of ether to evaporate spontaneously in a glass dish, the residue must have no odor, and should not redden or decolorize blue litmus paper; and it must be completely volatilized on warming on the water-bath.

Ethyl Peroxide, Hydrogen Peroxide, and Ozone. — On vigorously shaking 10 cc. of ether with 1 cc. of 1:10 aqueous potassium iodide solution in a completely filled glass-stoppered bottle, neither the ether nor the potassium iodide solution should acquire a color after standing one hour in the dark.

Aldehydes. — On covering pieces of potassium hydroxide the size of a pea with the ether, and setting aside in the dark for one half hour, the liquid must not acquire a yellow color.

Sulphur Compounds. — On shaking 20 cc. of ether with a globule of mercury for two minutes in a glass-stoppered bottle, the bright surface of the metal must not be tarnished; nor should a black precipitate form.

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* It must be remembered that ether is apt to absorb moisture from the air, particularly when poured from one vessel into another, acquiring in consequence a higher specific gravity.

Water. — On shaking 20 cc. of ether in a stoppered flask with 1 gm. of anhydrous copper sulphate, the latter should not acquire a green or blue color.

II

ETHER, ANHYDROUS, DISTILLED OVER SODIUM

TESTS OF PURITY

In addition to the tests given above, this preparation must answer the following requirement:

On introducing 15 cc. of ether into a dry, glass-stoppered flask with a freshly-cut piece of sodium the size of a pea, only a very slight evolution of gas should take place, and the bright metallic surfaces of the sodium should not completely lose their luster within six hours.

FURFURAL

(FURFUROL)

$C_5H_4O_2$. Mol. Wt. 96.03.

When freshly prepared, furfural is a clear, colorless liquid, which rapidly acquires a yellow color on exposure to light and air. Furfural has a specific gravity of 1.165 to 1.166, boils at 158 to 160° C., and is soluble in 12 parts of cold water. It is very easily soluble in alcohol and in ether.

NOTE. — Regarding the quantitative determination of furfural, see W. E. Stone, Ztschr. anal. Chem., **40**, 550 (1901); J. Anal. Appl. Chem., **5**, 421 (1895). W. Cormack, Ztschr. anal. Chem., **43**, 256 (1904); J. Chem. Soc., **77**, 990 (1900). Merck's Reagentien-Verzeichnis, p. 19 (1903); Lewkowitsch, Chem. Technol. and Anal. Oils, Fats, and Waxes, 3 ed.. Vol. II, p. 542 (1904). Merck's Reagentien-Verzeichnis, pp. 129, 157 (1903); H. Schiff, J. Chem. Soc., **52**, 571 (1887). Woltering, Pharm. Ztschr. f. Russl., **31**, 526 [Ztschr. anal. Chem., **36**, 410 (1897)].

GALLEIN, LIQUID

(GALLEIN)

A pale brown coloring matter, consisting of pyrogallolphthalein. Gallein in alcoholic solution is used as an indicator.

GLYCERIN

(GLYCEROL)

$C_3H_8O_3$. Mol. Wt. 92.06.

I

GLYCERIN, SP. GR. 1.250

A clear, colorless, odorless liquid, neutral in reaction, soluble in all proportions in water and in alcohol. Insoluble in ether and chloroform. Specific gravity not less than 1.250. It contains at least 95 per cent of absolute glycerin, $C_3H_8O_3$.

TESTS OF PURITY

Arsenic. — On mixing 1 cc. of glycerin with 3 cc. of stannous chloride solution, the mixture should not darken within one hour.

Free Acids and Bases. — On diluting 10 cc. of glycerin with 50 cc. of water, the solution should not affect red or blue litmus paper.

Inorganic Matter. — On boiling 5 cc. of glycerin in an open dish and then gently igniting, the glycerol should be volatilized completely, leaving only a dark stain which should disappear on being more strongly heated.

Substances which Reduce Ammoniacal Silver Nitrate Solution. — On heating a mixture of 1 cc. of glycerin and 1 cc. of ammonia water on the water-bath to 60° C., and then immediately adding 3 drops of silver nitrate solution, neither a coloration nor a brownish-black precipitate should develop within five minutes.

Fatty Acids. — On gently warming 1 cc. of glycerin with 1 cc. of 16 per cent sulphuric acid, no unpleasant, rancid odor should develop.

Hydrochloric Acid (Chlorides). — 5 cc. of glycerin diluted with 25 cc. of water should exhibit at most a slight, opalescent turbidity on the addition of silver nitrate solution.

Sulphuric and Oxalic Acids. — The solution of 5 gm. of glycerin in 25 cc. of water should not be affected by barium chloride solution nor by calcium chloride solution.

Heavy Metals. — On diluting 5 cc. of glycerin with 25 cc. of water and adding hydrogen sulphide water, no change should appear.

Calcium. — 5 cc. of glycerin dissolved in 25 cc. of water should not be affected by the addition of ammonium oxalate solution.

Sugars. — 5 cc. of glycerin are mixed with 50 cc. of water and a few drops of hydrochloric acid, heated thirty minutes on a water-bath, then 10 cc. of the hot liquid are made alkaline with sodium hydroxide and mixed with 1 cc. of Fehling's solution. No yellowish-red cloud or precipitate should appear within six hours.

Readily Carbonizable Matter. — 5 cc. of glycerin mixed with an equal volume of concentrated sulphuric acid, and allowed to stand one hour, should not become darker than yellow.

II

GLYCERIN, SP. GR. 1.23

A clear, colorless, odorless liquid of neutral reaction. Its specific gravity is 1.225–1.235.

TESTS OF PURITY

The tests for arsenic, heavy metals, chlorides, substances which reduce ammoniacal silver nitrate, free acids and bases, oxalic, sulphuric and fatty acids, and inorganic matter, are to be made in the manner described under Glycerin, sp. gr. 1.250.

The following additional test is also to be made:

Ammonium Compounds and Organic Bodies Such as Occur in Unpurified Glycerin. — On heating 1 cc. of glycerin with 1 cc. of sodium hydroxide solution, no ammonia should be evolved (to be detected by means of moistened litmus paper); nor should a color develop nor an odor resembling that of glue.

GUAIACIN ACCORDING TO SCHMITT *

Guaiacin is a brownish, amorphous powder, obtained by a special method from guaiac wood. It is soluble in alcohol, is colored blue by oxidizers, is far more sensitive than guaiac resin, and serves as an excellent reagent for oxidases. Guaiacin is used in the form of a 5 per cent alcoholic solution, which keeps well in completely filled, dark-colored, well-stoppered bottles.

HEMATEIN

$C_{16}H_{12}O_6$. Mol. Wt. 300.09.

Hematein occurs in reddish-brown plates, presenting a yellowish-green metallic luster. 100 parts of water at 20° C. dissolve 0.06 part of hematein. It is difficultly soluble in ether and in alcohol, and insoluble in chloroform and benzene; but it is soluble in ammonia water with brownish-violet color, and in dilute sodium hydroxide solution, producing a bright-red color.

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* Le Bois de Gajac, Thèse de Nancy. 1875. Report on the Advancements of Pharmaceutical Chemistry and Therapeutics, E. Merck, p. 73, 1902.

HEMATOXYLIN

$C_{16}H_{14}O_6 + 3H_2O$. Mol. Wt. 356.16.

Colorless, or pale-yellow, tetragonal prisms, melting at 100 to 120° C. with loss of water. On slowly cooling a solution saturated at boiling heat, hematoxylin crystallizes at times in the form of rhombic crystals containing one molecule of water of crystallization. Hematoxylin is but slightly soluble in cold water and in ether, but is more soluble in borax solution and in hot water, and is easily soluble in alcohol. On exposure to light it gradually acquires a red color, and then dissolves to form yellow solutions. In ammonia water hematoxylin dissolves and yields a purple solution. Hematoxylin is used as an indicator in 0.5 per cent alcoholic solution.

NOTE. — Regarding the use of hematoxylin as an indicator in the titration of alkaloids, see C. Kippenberger, Ztschr. anal. Chem., **39**, 201 (1900); J. Chem. Soc., **78**, II, 637 (1900). J. Messner, Ztschr. angew. Chem., **16**, 444 (1903); J. Chem. Soc., **84**, II, 519 (1903).

HIDE POWDER

White or yellowish-white, woolly powder, prepared from the best quality of hide from which the hair has been removed with lime and which has been thoroughly washed. Hide powder should have but a slight odor and should be specially free from odors of decomposition products. It is used for tannin determination.

TEST OF PURITY

Determination of the Water-Soluble Constituents.* — Introduce 5 gm. of the hide powder into a bell-filter, as described by Procter (see Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **3**, 715 (1905); Allen, Com. Organ. Anal., 3 ed., Vol. III, Pt. I, p. 83 (1900)). Then suspend the filter in a beaker of about 150 to 200 cc. capacity, fix the siphon, by means of a clamp, so that the bell-filter almost reaches the bottom of the vessel, and then pour into the beaker a small quantity of water in order to moisten the hide powder by capillarity. After the powder has become moist, which requires about an hour, the beaker is filled, and suction carefully applied to the siphon until the filtrate begins to drop slowly. The filtration of 90 to 100 cc. requires about one and a half to two hours. The 30 cc. first siphoned off are rejected; the next 50 cc. are evaporated to dryness on the water-bath. The residue from the evaporation is then dried to constant weight in an air-bath at 100 to 105° C. The weight of this residue, conforming to the requirements laid down by the International Conference of Chemists for Leather Industries, should not exceed 0.005 gm.

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* As the quantity of water-soluble constituents in the various hide powders varies greatly, it is always necessary, before using a hide powder for tannin determinations, to determine the quantity of soluble constituents by a blank test according to the method described here.

HYDROGEN PEROXIDE, 30%

(PERHYDROL)

H_2O_2. Mol. Wt. 34.01.

A colorless liquid of specific gravity 1.115 to 1.119, containing about 30 per cent by weight of hydrogen peroxide.* The solution is acid to litmus paper.†

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* The solution, containing 30 per cent, by weight, of hydrogen peroxide, is also designated as "100 volumes hydrogen peroxide," because this solution is capable of yielding 100 times its volume of free oxygen.

† The acid reaction is entirely due to the high content of hydrogen peroxide. If it is desired to ascertain whether the acidity is due also to the presence of acids, the hydrogen peroxide is first decomposed by means of a small piece of platinum sponge.

TESTS OF PURITY

Sulphuric Acid. — Dilute 1 cc. of the hydrogen peroxide with 20 cc. of water, add 0.5 cc. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Residue on Evaporation (Sulphuric and Phosphoric Acids, etc.). — 10 cc. of the hydrogen peroxide, when heated on the water-bath, should completely volatilize and leave no weighable residue.

Oxalic Acid. — The solution of 2 cc. of hydrogen peroxide in 10 cc. of water should not become turbid on the addition of calcium chloride solution.

Hydrochloric Acid. — The solution of 1 cc. of hydrogen peroxide in 20 cc. of water with 1 cc. of nitric acid should not become turbid on the addition of a few drops of silver nitrate solution.

Hydrofluoric Acid. — Evaporate 10 cc. of hydrogen peroxide with a few drops of sodium hydroxide solution, transfer the concentrated liquid to a watch glass, evaporate to dryness on the latter, pour concentrated sulphuric acid over the residue, and allow the whole to stand two to three hours in a warm place. After washing off the glass, no etching should be visible.

Phosphoric Acid. — Concentrate 5 cc. of hydrogen peroxide on the water-bath, take up any residue with 3 cc. of water, add 1 cc. of magnesia mixture and 3 cc. of ammonia water. No precipitation should take place on standing twelve hours.

Quantitative Determination. — Weigh off 1 gm. of hydrogen peroxide in a graduated flask of 100 cc. capacity, and fill with water to the mark. Dilute 20 cc. of this liquid with 50 cc. of water, add 40 cc. of 16 per cent sulphuric acid, and titrate with decinormal potassium permanganate. At least 35 cc. should be required to produce the end-point.

1 cc. of decinormal $KMnO_4$ = 0.0017008 gm. of H_2O_2, log. 23065.

HYDROGEN SULPHIDE WATER

A clear, colorless liquid, having a strong odor of hydrogen sulphide gas, and affording a voluminous precipitate of sulphur on the addition of ferric chloride solution. Hydrogen sulphide water has an acid reaction to litmus paper.

HYDROXYLAMINE HYDROCHLORIDE

$NH_2OH \cdot HCl$. Mol. Wt. 69.52.

Dry, colorless crystals, soluble in 1 part of water and in 15 parts of alcohol, and also in glycerin. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of hydroxylamine hydrochloride, heated on platinum foil, should volatilize and leave no residue.

Ammonium Chloride. — The 1:20 alcoholic solution should afford no precipitate on adding platinic chloride solution.

Sulphuric Acid. — On adding barium chloride solution to 20 cc. of a 1:10 aqueous solution of hydroxylamine hydrochloride, no precipitate of barium sulphate should form within twelve hours.

Iron. — 10 cc. of the 1:10 aqueous solution are boiled two or three minutes with 1 cc. of 15 per cent potassium hydroxide solution. While still hot, acidify with 1 cc. of strong nitric acid and subsequently add an excess of potassium sulphocyanate solution. No red color should appear.

Quantitative Determination. — Dissolve 1 gm. of hydroxylamine hydrochloride in 30 cc. of water, and titrate the solution with normal potassium hydroxide to permanent redness, using phenolphthalein as indicator.

1 cc. of normal KOH = 0.06952 gm. of $NH_2OH \cdot HCl$, log. 84211.

INDIGO

I

SYNTHETIC INDIGO

A fine, dark-blue powder, containing at least 95 per cent of indigo blue.

TESTS OF PURITY

Ash. — On heating 1 gm. of indigo in a platinum crucible, beautiful purple-red vapors are evolved, and a residue is left, the weight of which, after ignition, should not exceed 0.01 gm.

Moisture. — On drying 1 gm. of indigo at 100° C. to constant weight, the loss of weight should not exceed 0.01 gm.

Quantitative Determination of Indigo Blue (Indigotin *). — Place 1 gm. of indigo in a beaker of about 50 cc. capacity, cover it with 8 cc. of fuming sulphuric acid, containing 8 to 10 per cent of sulphuric anhydride, and warm the mixture on the sand-bath at 50 to 60° C., stirring frequently. The indigo will be dissolved in about one to two hours. Rinse the solution into a liter flask, and fill with water to the mark. Shake thoroughly, and transfer 100 cc. of the solution into a porcelain dish, add 400 cc. of water and 50 cc. of dilute sulphuric acid (1:10), then titrate immediately with decinormal potassium permanganate. At first no change in the dark-blue color is noted, but gradually the color passes through green into light yellow. This point (and not the

* A perfectly unobjectionable method of assaying indigo is not known at present. The method here described frequently gives results that are too high. The results, however, are satisfactory for fine grades of indigo, which rarely contain foreign organic compounds. In this method, however, indigo red is determined along with the indigo blue.

incipient red color) is the sharp and distinctly visible end-point of the titration.

1 cc. of decinormal $KMnO_4$ = 0.007415 gm. of indigotin, log. 87011.

II

VEGETABLE INDIGO

Dark-blue, light pieces, which, on being rubbed with a horn spoon, or other hard substances, exhibit a coppery metallic luster. Good vegetable indigo has a lower specific gravity than water, hence it floats on the latter. It should contain at least 60 per cent of indigo blue.

TESTS OF PURITY

Ash. — 1 gm. of indigo should not leave more than 0.12 gm. of ash.

Moisture. — On drying 1 gm. of indigo to constant weight at 100° C., the loss in weight should not exceed 0.06 gm.

Quantitative Determination of Indigo Blue (Indigotin). — This test is to be carried out as detailed under Synthetic Indigo.

INDIGOTIN

(INDIGO BLUE)

$C_{16}H_{10}N_2O_2$. Mol. Wt. 262.08.

Purple, rhombic crystals with coppery luster, or dark-blue powder with a reddish tint, which, on pressure or rubbing, exhibits a coppery luster. Indigo blue is insoluble in water, alcohol, ether, dilute acids, and alkalies. It dissolves in 15 parts of concentrated sulphuric acid, or 5 parts of fuming sulphuric acid, with the formation of indigo sulphonic acids.

TESTS OF PURITY

Ash. — 1 gm. of indigotin, on ignition, should yield a residue weighing not more than 0.0015 gm.

Moisture. — On drying 1 gm. of indigotin at 100° C. to constant weight, the loss of weight should not exceed 0.005 gm.

Quantitative Determination. — This is to be carried out as described under Synthetic Indigo.

IODEOSIN

(TETRAIODOFLUORESCEIN)

$C_{20}H_8I_4O_5$. Mol. Wt. 835.94.

A scarlet-red, crystalline powder, yielding with alcohol a deep-red, and with ether a yellowish-red, solution. Iodeosin is insoluble in water containing a trace of hydrochloric acid. The solution used as an indicator is prepared by dissolving 1 gm. of iodeosin in 500 cc. of alcohol.

TEST OF SENSITIVENESS

Introduce 100 cc. of water, containing 5 drops of the above iodeosin solution, into a colorless, glass-stoppered flask, and then overlay it with 30 cc. of ether. Then run in by drops, from a burette, centinormal hydrochloric acid solution, vigorously shaking after the addition of each drop, until the aqueous layer just becomes colorless. Now add 5 drops more of the iodeosin solution. After shaking again, the aqueous layer must not acquire a pink color; or, should it do so, the color must disappear on the addition of 1 drop of centinormal hydrochloric acid.

NOTE. — Regarding the use of iodeosin for the estimation of alkaloids, see C. Kippenberger, Ztschr. anal. Chem., **39**, 205 (1900); J. Chem. Soc., **78**, II, 637 (1900). Merck's Berichte, pp. 9, 32 (1900); Merck's Annual Report, pp, 13, 37 (for the year 1900).

IODINE

(IODINE RESUBLIMED)

I. Atomic Wt. 126.97.

Blackish-gray, dry, rhombic plates or scales, with a metallic luster, easily soluble in alcohol, ether, and chloroform. Iodine dissolves in about 5000 parts of water, and is very freely soluble in an aqueous solution of potassium iodide. The preparation contains 99.98 to 100 per cent of I.

TESTS OF PURITY

Non-volatile Matter. — On cautiously heating 1 gm. of iodine in a porcelain dish, no weighable residue should remain.

Cyanogen, Chlorine, and Bromine. — Shake 0.5 gm. of powdered iodine with 20 cc. of water, and filter. To 10 cc. of the filtrate, add, drop by drop, decinormal sodium thiosulphate solution until decolorized, then add a granule of ferrous sulphate, 1 drop of ferric chloride solution and 2 cc. of sodium hydroxide solution. Warm to about 60° C., and add 10 cc. of hydrochloric acid. The liquid should not acquire a blue color. Filter, and to 10 cc. of the filtrate add 1 cc. of ammonia water, 5 drops of silver nitrate solution, and again filter. On adding to the filtrate 2 cc. of nitric acid no precipitate, and not more than an opalescent turbidity, should develop.*

Quantitative Determination. — Dissolve 0.2 gm. of iodine with 1 gm. of potassium iodide in 50 cc. of water, and titrate with decinormal sodium thiosulphate.†

1 cc. of decinormal $Na_2S_2O_3$ = 0.012697 gm. of I, log. 10369.

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* The filtrate contains traces of dissolved silver iodide; hence, on acidulating with nitric acid some opalescence always occurs.

† The titer of the decinormal sodium thiosulphate should be controlled by means of potassium biniodate.

IODINE WATER

A saturated aqueous solution, containing about 0.2 gm. of iodine in 1000 cc.

TEST OF STRENGTH

Titrate 100 cc. of iodine water with decinormal sodium thiosulphate solution.

1 cc. of decinormal $Na_2S_2O_3$ = 0.012697 gm. of I, log. 10369.

IRON

Fe. Atomic Wt. 55.9.

Metallic iron is used in the analytical laboratory in the form of thin, polished wire (piano wire), or gray powder obtained by reduction with hydrogen (iron by hydrogen), and also in the form of the ordinary gray, lustrous powdered iron.

I

IRON WIRE

(PIANO WIRE)

Iron wire serves chiefly for the standardization of potassium permanganate solutions. It was formerly believed, without further consideration, that the wire contained 99.6 to 99.8 per cent of pure, active iron. Treadwell, however, has shown that the various kinds of wire exhibit a decidedly variable value towards potassium permanganate solution, and that several exhibit a value corresponding to over 100 per cent of Fe, which statement is fully confirmed by Lunge. The explanation of this lies in the presence of carbon in the wire, principally as carbide of iron, which, upon dissolving in acid, becomes converted into hydrocarbons, exerting a marked reducing action on the permanganate.

Notwithstanding this, iron wire is a convenient, unalterable, and also perfectly reliable substance for standardizing permanganate solutions, if we have once ascertained its effective value compared with potassium permanganate solution, the titer of which has been accurately determined by means of some other standard.

For a satisfactory means of fixing the titer of permanganate solution, sodium oxalate may be recommended (see Sodium Oxalate, According to Sörensen). Or * starting with sodium bicarbonate, then by drying at about 300° C., thus obtaining sodium carbonate, preparing a standard solution of this, from this preparing a solution of fifth normal hydrochloric acid, from this a solution of fifth normal potassium hydroxide, from this a solution of oxalic acid, and finally a solution of the permanganate. Where a gasvolumeter is at hand, the titer may also be ascertained with hydrogen peroxide, according to the nitrometer method. (G. Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **1**, 127 (1904); J. Chem. Soc., **58**, 1468 (1890).)

II

IRON BY HYDROGEN

(Reduced Iron)

A gray, lusterless, fine powder, containing at least 90 per cent of Fe.

TESTS OF PURITY

Solubility in Acids; Carbon and Silica. — 10 gm. of reduced iron should dissolve almost completely in a mixture of 20 cc. of sulphuric acid (sp. gr. 1.84) and 200 cc. of water. Any insoluble residue should be filtered off, and when dried at 100° C. its weight should not exceed 0.05 gm.

Sulphides. — Pour a mixture of 10 cc. of hydrochloric acid and 10 cc. of water over 1 gm. of reduced iron in a test tube. Filter paper moistened with solution of lead acetate should not be darkened in color on exposure to the liberated hydrogen for ten seconds.

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* G. Lunge, Ztschr. angew. Chem. **17**., 267, (1904); J. Chem. Soc., **86** II, 289 (1904).

Sodium Carbonate. — On shaking 5 gm. of reduced iron with 50 cc. of water and filtering, the filtrate should not turn red litmus paper blue; and on evaporation it should leave no residue.

Nitrogen. — Dissolve 10 gm. of reduced iron in a mixture of 20 cc. of concentrated sulphuric acid (sp. gr. 1.84) and 200 cc. of water, with the aid of heat. Allow to cool, and when cold add 100 cc. of sodium hydroxide solution (sp. gr. 1.3), and distil off about 50 cc., collecting the distillate in a receiver containing about 20 cc. of water and 2 to 3 cc. of decinormal hydrochloric acid. Titrate the distillate with decinormal potassium hydroxide, using methyl orange as indicator. The ammonia should not have neutralized more than 0.2 cc. of the acid.

Arsenic. — Pour 10 cc. of hydrochloric acid over a mixture of 1 gm. of reduced iron and 1 gm. of potassium chlorate, and when the reaction has subsided, heat until all free chlorine has been expelled. On now adding 15 cc. of stannous chloride solution to 5 cc. of the filtrate, a dark coloration should not develop within one hour.

Quantitative Determination. — Introduce into a graduated flask of 100 cc. capacity 1 gm. of reduced iron, 10 gm. of finely powdered mercuric chloride, and 50 cc. of boiling water. Place the flask on wire gauze, heat over a small flame, boil for about five minutes with frequent shaking; then immediately fill the flask to the mark with cold, boiled water. Cool the mixture to 15° C., add water to the mark, shake thoroughly, and, tightly stoppering the flask, set it aside to deposit. Now filter, and to 10 cc. of the filtrate add immediately 10 cc. of 16 per cent sulphuric acid, and titrate with decinormal potassium permanganate until a faint red color persists. At least 16 cc. of the permanganate solution should be required.

For the purpose of control, dissolve in the slightly red liquid 2 gm. of potassium iodide, allow to stand for one hour in the stoppered flask at 20° C., and titrate with decinormal sodium thiosulphate solution, using starch solution as the indicator.

1 cc. of decinormal $KMnO_4$ or 1 cc. of decinormal $Na_2S_2O_3$ = 0.00559 gm. of Fe, log. 74741.

III

IRON POWDER

A fine, heavy, gray powder with metallic luster, and containing at least 98 per cent of Fe.

TESTS OF PURITY

Solubility in Acids; Carbon, Silica, and Sulphur. — 10 gm. of powdered iron should dissolve almost completely in a mixture of 20 cc. of concentrated sulphuric acid (sp. gr. 1.84) and 200 cc. of water. Any insoluble residue should be filtered off and dried at 100° C. Its weight should not exceed 0.005 gm. The gas evolved during solution should not impart more than a brownish color within five seconds to strips of filter paper moistened with lead acetate solution.

Nitrogen and Arsenic. — These tests are carried out as detailed under Iron by Hydrogen.

Copper, Zinc, and Lead. — Dissolve 1 gm. of powdered iron in 25 cc. of nitric acid, assisting solution by heating the mixture to boiling. Dilute the solution with 25 cc. of water, add 30 cc. of ammonia water, and filter. The filtrate should not have a blue color; nor should it show any change on the addition of hydrogen sulphide water.

Quantitative Determination. — Dissolve 1 gm. of powdered iron in about 50 cc. of dilute sulphuric acid, and dilute the solution to 100 cc. To 10 cc. of this solution add decinormal potassium permanganate solution until the liquid has a slight red color, and when the liquid becomes decolorized, which may be effected, if necessary, by adding a few drops of alcohol, add 2 gm. of potassium iodide. Allow the mixture to stand one hour in a closed flask at 20° C., and then titrate with decinormal sodium thiosulphate. At least 17.5 cc. should be required to combine with the liberated iodine.

1 cc. of decinormal $Na_2S_2O_3$ = 0.00559 gm. of Fe, log. 74741.

IRON CHLORIDE, FERRIC

$FeCl_3 + 6H_2O$. Mol. Wt. 270.34.

Yellow, crystalline masses, very deliquescent in air. Ferric chloride is easily soluble in water, alcohol, and a mixture of alcohol and ether. The solutions are acid to litmus paper.

TESTS OF PURITY

Basic Salt and other Material Difficultly Soluble in Water. — 10 gm. of ferric chloride should completely dissolve in 10 cc. of water, and yield a perfectly clear solution.

Hydrochloric Acid and Chlorine. — On bringing a glass rod, moistened with ammonia water, over a few cubic centimeters of a ferric chloride solution (1:1), contained in a watch glass, no cloud should form. On introducing a piece of paper, moistened with zinc iodide-starch solution, into the neck of a flask containing (1:1) ferric chloride solution, the paper should not acquire a blue color within two minutes.

Arsenic. — A mixture of 1 cc. of ferric chloride solution (1:1) and 3 cc. of stannous chloride solution should not acquire a darker color within one hour.

Ferrous Salt. — Add 1 cc. of hydrochloric acid and a few drops of potassium ferricyanide solution to the 1:20 aqueous solution of ferric chloride; no blue color should develop.

Copper, Nitric Acid, etc. (Alkali Salts, Calcium). — Dilute 20 cc. of ferric chloride solution (1:1) with 100 cc. of water, add 25 cc. of ammonia water, and filter. On evaporating 50 cc. of the colorless filtrate and igniting the residue, the weight of the latter should not exceed 0.001 gm. On mixing 2 cc. of the filtrate with 2 cc. of concentrated sulphuric acid, and overlaying this mixture with 1 cc. of ferrous sulphate solution, no brown zone should form at the contact-surfaces of the two liquids. 20 cc. of the filtrate acidulated with acetic acid should not be affected by potassium ferrocyanide solution.

Sulphates. — Dissolve 10 gm. of ferric chloride in 100 cc. of water, add 25 cc. of ammonia water, filter, acidulate the filtrate with acetic acid, and add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

IRON CHLORIDE, FERRIC, SOLUTION

A clear, deep yellowish-brown liquid, of specific gravity 1.280 to 1.282. 100 parts contain 10 parts of Fe = 29 parts of $FeCl_3$.

TESTS OF PURITY

The tests to be made are those given under Iron Chloride, Ferric. But, for each gram of the crystallized ferric chloride, 3 cc. of the solution of ferric chloride are to be taken.

IRON CHLORIDE, FERROUS

$FeCl_2 + 4H_2O$. Mol. Wt. 198.86.

A pale-green hygroscopic powder, soluble in an equal weight of water acidulated with a few drops of hydrochloric acid, yielding a pale-green solution. Iron chloride is also soluble in alcohol.

TESTS OF PURITY

Oxychloride. — The solution of 1 gm. of ferrous chloride in 1 cc. of water and 2 to 3 drops of hydrochloric acid should be pale green or green in color, and should not have any yellowish-green tint. On adding 5 cc. of saturated hydrogen sulphide water, the solution should show only a very slight turbidity, due to the separation of sulphur.*

Sulphates, Copper, and Alkalies. — Boil for a few minutes a solution of 5 gm. of ferrous chloride in 10 cc. of water and 5 cc. of nitric acid (sp. gr. 1.3), dilute to 120 cc., add 20 cc. of ammonia water, and filter; evaporate 50 cc. of the filtrate and ignite the residue. The weight of the latter should not exceed 0.001 gm. Slightly acidulate 20 cc. of the filtrate with hydrochloric acid and add barium nitrate solution. No change should appear. 20 cc. of the filtrate acidified with acetic acid should show no change upon addition of potassium ferrocyanide solution.

Arsenic. — The solution of 1 gm. of ferrous chloride in 1 cc. of water, acidulated with a few drops of hydrochloric acid, should not acquire a darker color on adding 3 cc. of stannous chloride solution and standing one hour.

IRON SULPHATE, FERROUS

$FeSO_4 + 7H_2O$. Mol. Wt. 278. 07.

Pale, greenish-blue, monoclinic crystals, soluble in 1.8 parts of cold water and in 0.5 part of boiling water; insoluble in alcohol and in ether.

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* Small amounts of iron oxychloride are always apt to be present in ferrous chloride, even when the latter is most carefully made.

TESTS OF PURITY

Substances Insoluble in Water. — The 1:20 solution, freshly prepared with water which has previously been thoroughly boiled and then cooled, should be clear and have a greenish-yellow color.

Alkalies. — Add 5 cc. of nitric acid (sp. gr. 1.3) to a solution of 5 gm. of ferrous sulphate in 100 cc. of water, and boil for several minutes; then add 15 cc. of ammonia water, filter, and evaporate the filtrate. The residue ignited should not exceed 0.001 gm. in weight.

Copper and Zinc. — Oxidize the solution of 2 gm. of ferrous sulphate in 50 cc. of water by boiling with 3 cc. of nitric acid (sp. gr. 1.3), then add 8 cc. of ammonia water and filter:

(*a*) 15 cc. of the filtrate should undergo no change on the addition of a few drops of ammonium sulphide solution.

(*b*) Acidify 20 cc. of the filtrate with acetic acid, and add potassium ferrocyanide solution; the liquid should remain unchanged.

Quantitative Determination. — Dissolve 1 gm. of ferrous sulphate in 50 cc. of well-boiled water, add 10 cc. of dilute sulphuric acid, and titrate with decinormal potassium permanganate.

1 cc. of decinormal $KMnO_4$ = 0.027807 gm. of $Fe\,SO_4 + 7H_2O$, log. 44415.

IRON SULPHIDE

(FERROUS SULPHIDE)

FeS. Mol. Wt. 87.96.

Dark gray or grayish-black, heavy, hard lumps, soluble in diluted hydrochloric or sulphuric acid with the copious evolution of hydrogen sulphide.

TEST OF PURITY

Quantitative Determination. — Place 0.5 gm. of the finely powdered iron sulphide in a retort, in the tubule of which is fixed a funnel-tube provided with a glass cock. After connecting the retort with a receiver containing 50 to 100 cc. of decinormal iodine solution, allow to flow into the retort, through the funnel-tube, a mixture of 20 cc. of water and 20 cc. of dilute sulphuric acid; close the stop-cock, and heat to boiling. After the decomposition of the iron sulphide is complete, and the hydrogen sulphide has been entirely driven off (the iodine solution must not be fully decolorized), determine the excess of iodine by means of decinormal sodium thiosulphate.

1 cc. of decinormal I = 0.004398 gm. of FeS, log. 64326.

IRON AND AMMONIUM SULPHATE, FERRIC

(FERRIC AMMONIUM SULPHATE; AMMONIO-FERRIC ALUM)

$FeNH_4(SO_4)_2 + 12H_2O$. Mol. Wt. 482.28.

Large, pale violet (amethyst colored), transparent, octahedral crystals, soluble in 2 parts of water and insoluble in alcohol. The aqueous solution has an acid reaction.

TESTS OF PURITY

Ferrous Salt. — Dissolve 1 gm. of ferric ammonium sulphate in 20 cc. of water, add 1 cc. of hydrochloric acid and 1 drop of a freshly prepared solution of potassium ferricyanide. Neither a green nor a blue color should develop.

Chlorides. — 30 cc. of the 1:20 aqueous solution should undergo no change on the addition of 3 cc. of nitric acid and silver nitrate solution.

Zinc and Copper. — To a solution of 2 gm. of ferric ammonium sulphate in 50 cc. of water add 10 cc. of ammonia water and filter. The filtrate should be colorless.

(*a*) 20 cc. of the filtrate should show no change on adding ammonium sulphide solution.

(*b*) On acidifying 20 cc. of the filtrate with acetic acid, and then adding potassium ferrocyanide solution, the liquid should show no change.

Alkalies. — To a solution of 5 gm. of ferric ammonium sulphate in 100 cc. of water add 15 cc. of ammonia water, filter, and evaporate the filtrate. The residue ignited should not exceed 0.002 gm. in weight.

IRON AND AMMONIUM SULPHATE, FERROUS

(Ferrous Ammonium Sulphate; Mohr's Salt)

$FeSO_4(NH_4)_2SO_4 + 6H_2O$. Mol. Wt. 392.26.

Pale, bluish-green crystals, or light, bluish-green crystalline powder, soluble in 6 parts of cold water. Ferrous ammonium sulphate contains exactly one seventh of its weight of Fe in the ferrous condition.

TESTS OF PURITY

Ferric Salts. — The solution of 1 gm. of powdered ferrous ammonium sulphate in 20 cc. of boiled and cooled water (freed from oxygen), with 1 cc. of hydrochloric acid, should not afford an immediate red color on adding a few drops of potassium sulphocyanate solution.

Copper, Zinc, and Alkali Salts. — Dissolve 5 gm. of ferrous ammonium sulphate in 50 cc. of water, add 5 cc. of nitric acid (sp. gr. 1.3), and boil the solution for a few minutes; then add to the liquid 15 cc. of ammonia water, let stand for about one hour, and filter. The filtrate must be colorless (copper).

Add hydrogen sulphide water to 10 cc. of the filtrate; no change in appearance should occur (zinc).

Evaporate 30 cc. of the filtrate on the water-bath to dryness and ignite. No weighable residue should remain.

Quantitative Determination. — Dissolve 1 gm. of ferrous ammonium sulphate in 50 cc. of well-boiled and cooled water, add 10 cc. of dilute sulphuric acid, and titrate with decinormal potassium permanganate solution.

1 cc. of decinormal $KMnO_4$ = 0.039226 gm. of $FeSO_4(NH_4)_2SO_4 + 6H_2O$, log. 59358.

LACMOID

(Resorcinol Blue)

Lustrous, dark-violet scales, soluble in alcohol, acetone, and ether, and but slightly soluble in water. The solution to be used as an indicator is prepared by dissolving 0.5 gm. of lacmoid in 100 cc. of 95 per cent alcohol, and diluting the solution with 100 cc. of water.

TESTS OF SENSITIVENESS

(*a*) Introduce 100 cc. of absolute alcohol into a flask provided with a glass stopper, and then add 0.2 cc. of the above lacmoid solution; the blue color of the alcohol should change to red on the addition of 0.05 cc. of decinormal hydrochloric acid, and the red color should again change to blue on the further addition of 0.05 cc. of decinormal potassium hydroxide.

(*b*) Add 0.2 cc. of the above lacmoid solution to 100 cc. of distilled water freed from carbon dioxide by prolonged boiling in a platinum dish; the blue color of the water must change to red on the addition of 0.05 cc. of decinormal hydrochloric acid, and the red color should again become blue on the further addition of 0.05 cc. of decinormal potassium hydroxide.

NOTE. — Regarding the use of lacmoid for the titration of alkaloids, see C. Kippenberger, Ztschr. anal. Chem., **39**, 214 (1900); J. Chem. Soc., **78**, II, 637 (1900). J. Messner, Ztschr. angew. Chem., **16**, 444 (1903); J. Chem. Soc., **84**, II, 519 (1903).

LEAD ACETATE

$Pb(C_2H_3O_2)_2 + 3H_2O$. Mol. Wt. 378.99.

Colorless, translucent crystals, soluble in 2.3 parts of water or in 29 parts of 85 per cent alcohol. All the aqueous solutions for testing should be prepared with well-boiled water, to insure freedom from carbon dioxide.

TESTS OF PURITY

Earths and Alkalies. — Dissolve 5 gm. of lead acetate in about 100 cc. of water, and precipitate the lead from the solution with hydrogen sulphide gas; filter, evaporate the filtrate, and ignite the residue. The weight of the latter should then not exceed 0.001 gm.

Copper and Iron. — Dissolve 2 gm. of lead acetate in 40 cc. of water, add to the solution 50 cc. of ammonia water and, as soon as the precipitate has settled, filter. The precipitate must have a pure white color, and the filtrate should be colorless.

Lead Carbonate and Substances Insoluble in Water. — The solution of 5 gm. of lead acetate in 50 cc. of water should be clear, or only very slightly opalescent.

Chlorides. — The 1:30 aqueous solution acidified with nitric acid should show no change with silver nitrate solution.

Nitrates. — The solution of 1 gm. of lead acetate in 30 cc. of water, which has been colored a light blue by the addition of 1 drop of indigo solution, should not lose its color on the addition of 15 cc. of concentrated sulphuric acid.

LEAD CHROMATE

$PbCrO_4$. Mol. Wt. 323.

A yellowish-brown powder, or brown lumps, insoluble in water and ammonia water; but almost completely soluble in solutions of the fixed alkali hydroxides, and, with decomposition, almost wholly soluble also in concentrated nitric acid.

TESTS OF PURITY

Substances Soluble in Water. — Shake 5 gm. of lead chromate in fine powder with 50 cc. of warm water (at about 50°C.) for five minutes, then filter, evaporate the filtrate, and ignite the residue. The weight of the latter should not exceed 0.001 gm.

Organic Substances. — Lead chromate should evolve no carbon dioxide on ignition.

Lead chromate which is to be used in the elementary analysis of organic compounds should first be gently ignited in a current of oxygen, as according to C. H. L. Ritthausen [J. prakt. Chem., N. F., **25**, 141 (1882); J. Chem. Soc., **42**, 898 (1882)] when ignited in air, it retains carbon, which can only be burned off by ignition in a current of oxygen.

LEAD OXIDE, BROWN

(LEAD SUPER-, DI-, OR PEROXIDE)

PbO_2. Mol. Wt. 238.9.

I

LEAD OXIDE BROWN, FREE FROM MANGANESE

A dark-brown, amorphous powder, insoluble in water. The preparation contains 97.5 to 99 per cent of PbO_2.

TESTS OF PURITY

Chlorides. — Boil 5 gm. of lead peroxide with 60 cc. of water and 5 cc. of nitric acid, filter, and to 30 cc. of the filtrate add silver nitrate solution. At most a slight opalescent turbidity should appear.

Sulphates. — Digest 5 gm. of lead peroxide with 30 cc. of a cold saturated aqueous solution of sodium bicarbonate for three or four hours, shaking frequently. Then filter, acidulate the filtrate with hydrochloric acid, boil the solution for ten minutes, and add 2 cc. of barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Substances Soluble in Water (Lead Nitrate, etc.). — Boil 2 gm. of lead peroxide with 60 cc. of water, and filter; 40 cc. of the filtrate should not leave a weighable residue upon evaporation and ignition.

Calcium and Alkalies. — Dissolve 2 gm. of lead peroxide in 25 cc. of hydrochloric acid. Dilute the liquid with 200 cc. of water, boil ten minutes to expel the chlorine, and supersaturate with hydrogen sulphide gas. Filter off the precipitate, evaporate the filtrate, ignite and weigh the residue. The weight should not exceed 0.002 gm.

Manganese. — Heat 5 gm. of lead peroxide with 10 cc. of concentrated sulphuric acid until completely decomposed. When cold, mix the mass with 20 cc. of water and add 0.5 gm. of lead peroxide. On now warming again, the liquid should not acquire a red color.

Quantitative Determination. — Gently heat 1 gm. of lead peroxide in a porcelain dish of about 300 cc. capacity with 30 cc. of nitric acid; then add, while stirring, 30 cc. of seminormal oxalic acid solution, and heat to 60° C. The lead peroxide must be completely dissolved by this treatment, and the solution should be clear and colorless. Now heat the solution to the boiling point, and allow semi-normal potassium permanganate solution to run into it until the red color imparted by 1 drop of the permanganate solution no longer completely disappears within half a minute. 13.5 to 13.7 cc. of the semi-normal potassium permanganate solution should be required.

1 cc. of semi-normal $H_2C_2O_4$ = 0.05973 gm. of PbO_2, log. 77619.

II

LEAD OXIDE BROWN, FOR DENNSTEDT'S ULTIMATE ANALYSIS

A dark-brown, amorphous powder, insoluble in water, used for ultimate organic analysis according to Dennstedt.*

TESTS OF PURITY

Chlorides; Calcium and Alkalies. — The tests are to be carried out as detailed under lead peroxide free from manganese.

Sulphates. — Digest 25 gm. of lead peroxide with 50 cc. of a cold saturated aqueous solution of sodium bicarbonate for three to four hours, shaking frequently. Then filter, acidulate the filtrate with hydrochloric acid, boil the solution for ten minutes, and add 2 cc. of barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Nitrates. — Boil 1 gm. of lead peroxide with a mixture of 5 cc. of dilute acetic acid and 10 cc. of water, filter, and color the filtrate blue by adding 1 drop of a 1:1000 indigo solution. The blue color should not disappear on the addition of 10 cc. of concentrated sulphuric acid.

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* Literature: M. Dennstedt, Ztschr. anal. Chem., **41**, 525 (1902); J. Chem. Soc., **84**, II, 103 (1903); Also: Anleitung zur vereinfachten Elementaranalyse, von Prof. Dr. M. Dennstedt, 1903. Published by O. Meissner, Hamburg.

Carbonates. — On pouring nitric acid over 5 gm. of lead peroxide, no evolution of gas should be seen, even when observed through a magnifying glass.

Quantitative Determination. — This is to be carried out as detailed under Lead Peroxide Free From Manganese.

LEAD OXIDE YELLOW

(LITHARGE)

PbO. Mol. Wt. 222.9.

A yellow or reddish-yellow powder, almost insoluble in water (1:12000), but soluble in nitric acid, acetic acid, and potassium hydroxide solution.

TESTS OF PURITY

Substances Insoluble in Acetic Acid. — To 2 gm. of lead oxide mixed with 5 cc. of water add 10 cc. of dilute acetic acid. No evolution of gas should take place. Now boil the liquid for several minutes, filter when cold, wash the undissolved residue, dry at 100° C., and weigh. The weight should not exceed 0.005 gm.

Carbonates. — 5 gm. of lead oxide, when heated even to the melting-point, should not lose more than 0.005 gm. in weight.

Copper and Alumina. — Dissolve 2 gm. of lead oxide in 10 cc. of nitric acid and 5 cc. of water with the aid of heat. Add to the clear solution 15 cc. of 16 per cent sulphuric acid, and filter off the resulting precipitate after it has settled. On adding to the filtrate an excess of ammonia water, the liquid should not acquire a blue color, nor should a precipitate form.

Nitrates and Nitrites. — Mix 1 gm. of lead oxide with 5 cc. of water, and dissolve with the aid of 5 cc. of dilute acetic acid. The blue color produced by adding 1 drop of indigo solution should not disappear on the addition of 10 cc. of concentrated sulphuric acid.

Chlorides. — The solution of 1 gm. of lead oxide in 5 cc. of nitric acid diluted with 20 cc. of water should not be affected by silver nitrate solution.

Earths, Gypsum, and Alkalies. — Dissolve 1 gm. of lead oxide in 10 cc. of diluted acetic acid, add 100 cc. of water, and supersaturate with hydrogen sulphide gas. Filter off the precipitate, evaporate the filtrate, ignite the residue, and weigh. The weight should not exceed 0.003 gm.

LEAD SUBACETATE SOLUTION

A clear, colorless, liquid, of specific gravity 1.235 to 1.240. Lead subacetate solution is alkaline to litmus paper, but does not redden phenolphthalein.

TEST OF PURITY

Copper and Iron. — On acidifying 10 cc. of lead subacetate solution with 2 cc. of dilute acetic acid, and adding potassium ferrocyanide solution, a precipitate forms which should have a pure white color.

LIME, CHLORINATED

White cubes with chlorine-like odor, and liberating chlorine on the addition of hydrochloric acid. 100 parts treated with hydrochloric acid should afford at least 25 parts of Cl. Specially prepared in cube form for the generation of chlorine in the laboratory.

TEST OF PURITY

The value of this preparation depends entirely upon its active chlorine.

Quantitative Determination of Active Chlorine. — Mix 0.5

gm. of the powdered cubes with a solution of 3 gm. of potassium iodide in 60 cc. of water, and add 5 cc. of hydrochloric acid. Titrate the liberated iodine with decinormal sodium thiosulphate solution, of which at least 35.2 cc. should be required.

1 cc. of decinormal $Na_2S_2O_3$ = 0.003545 gm. of Cl, log. 54962.

LIME WATER

(Solution of Calcium Hydroxide)

A clear, colorless liquid having a strongly alkaline reaction.

TEST OF STRENGTH

Titrate 100 cc. of lime water with normal hydrochloric acid, using methyl orange as the indicator. Not less than 4 cc., nor more than 4.5 cc., of the normal hydrochloric acid should be required to produce the red end-point.

1 cc. of normal HCl = 0.03705 gm. of $Ca(OH)_2$, log. 56879.

LITMUS

A specially purified dye obtained from various species of lichens. It occurs in the form of friable lumps consisting chiefly of erythrolitmin and azolitmin.

A large number of formulas have been given for the preparation of litmus tincture, of which several are described by Glaser in his "Indikatoren der Azidimetrie und Alkalimetrie." A very sensitive litmus tincture may be prepared as follows:

Dissolve 1 gm. of litmus in 100 cc. of hot water, and add, drop by drop, dilute sulphuric acid until the liquid acquires a red color. Then boil for ten minutes in order to expel completely the carbon dioxide. Should the red color pass into blue during the boiling, the color is restored by again adding dilute sulphuric acid. Now add baryta water, drop by drop, until a violet color develops, set aside to deposit, and filter. Preserve the litmus tincture in bottles not completely filled, and simply cover with a loose cap, or stopper with a plug of cotton to keep out the dust.

TEST OF SENSITIVENESS

Add 0.2 cc. of litmus tincture to 100 cc. of distilled water, and boil for some time in a platinum dish to free it from carbon dioxide. The violet color of the liquid so obtained must change to red on the addition of 0.05 cc. of decinormal hydrochloric acid; or must pass into a pure blue color on the addition of 0.05 cc. of decinormal potassium hydroxide.

MAGNESIUM CARBONATE

$MgCO_3$. Mol. Wt. 84.36.

White, light pieces, easily reduced to powder; or white, bulky powder. Almost insoluble in water, it yet imparts to it a slight alkaline reaction.

TESTS OF PURITY

Substances Soluble in Water. — Suspend 5 gm. of powdered magnesium carbonate in 50 cc. of water, heat to boiling, and filter while hot. Evaporate 20 cc. of the filtrate, and weigh the residue, which should not exceed 0.006 gm.

Substances Insoluble in Hydrochloric Acid. — 1 gm. of the carbonate should entirely dissolve in 10 cc. of hydrochloric acid with 10 cc. of water, and yield a colorless solution.

Chlorides. — Dissolve 1 gm. of magnesium carbonate in 5 cc. of nitric acid and 15 cc. of water; on adding silver nitrate solution, not more than a slight opalescent turbidity should develop.

Sulphates. — On adding barium chloride solution to the solution of 1 gm. of magnesium cabornate in 5 cc. of hydrochloric acid and 15 cc. of water, no change should occur.

Barium. — The solution of 1 gm. of magnesium carbonate in 5 cc. of hydrochloric acid and 15 cc. of water should not be rendered turbid on the addition of dilute sulphuric acid.

Calcium and Alumina. — Dissolve 1 gm. of magnesium carbonate in 30 cc. of acetic acid (sp. gr. 1.041) and 30 cc. of water. Boil, add 20 cc. of ammonia water and some ammonium oxalate solution. The liquid should not become turbid within five minutes.

Heavy Metals. — The solution of 1 gm. of magnesium carbonate in 20 cc. of acetic acid (sp. gr. 1.041) and 30 cc. of water should be clear, and should show no change with hydrogen sulphide water.

Iron. — The solution of 1 gm. of magnesium carbonate in 5 cc. of hydrochloric acid and 15 cc. of water should not immediately acquire a blue color on the addition of 0.5 cc. of potassium ferrocyanide solution.

MAGNESIUM CHLORIDE

$MgCl_2 + 6H_2O$. Mol. Wt. 203.35.

White, deliquescent crystals, soluble in about 0.6 part of cold, or in 0.3 part of hot, water, and in 5 parts of 85 per cent alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Alcohol. — 2 gm. of magnesium chloride should completely dissolve in 10 cc. of 85 per cent alcohol, and yield a clear solution.

Sulphates. — The solution of 1 gm. of magnesium chloride in 20 cc. of water, slightly acidulated with hydrochloric acid, should not become turbid on the addition of barium chloride solution.

Phosphates and Arsenates. — Dissolve 3 gm. of magnesium chloride and 6 gm. of ammonium chloride in 24 cc. of water, and add 12 cc. of ammonia water. No turbidity or precipitate should form on standing twelve hours.

Ammonium Salts. — On heating 3 gm. of magnesium chloride with 10 cc. of sodium hydroxide solution (sp. gr. 1.3), no vapors of ammonia should be evolved (to be ascertained by moistened litmus paper).

Barium. — The solution of 1 gm. of magnesium chloride in 20 cc. of water should afford no reaction on the addition of dilute sulphuric acid.

Heavy Metals and Calcium.

(*a*) The solution of 1 gm. of magnesium chloride in 20 cc. of water must not be affected by hydrogen sulphide water.

(*b*) Dissolve 1 gm. of magnesium chloride in 20 cc. of water, and add 1 gm. of ammonium chloride and 5 cc. of ammonia water; no change should occur on the further addition of ammonium oxalate solution, nor with a few drops of ammonium sulphide solution.

MAGNESIUM OXIDE

(MAGNESIA)

MgO. Mol. Wt. 40.36.

I

MAGNESIUM OXIDE

A white, light, fine powder, almost insoluble in water.

TESTS OF PURITY

Substances Soluble in Water. — Heat 0.5 gm. of magnesium oxide with 20 cc. of water to boiling, allow to cool, and filter off 10 cc. of the supernatant liquid. The filtrate should have at most a slight alkaline reaction, and on evaporation should not yield a residue weighing more than 0.002 gm.

Substances Insoluble in Hydrochloric Acid. — 1 gm. of magnesium oxide should completely dissolve in 10 cc. of hydrochloric acid and 10 cc. of water, yielding a clear solution.

Sulphates. — The solution of 0.5 gm. of magnesium oxide in 10 cc. of acetic acid (sp. gr. 1.041) should not be immediately rendered turbid on the addition of barium nitrate solution.

Chlorides. — The solution of 0.5 gm. of magnesium oxide in 10 cc. of nitric acid and 10 cc. of water should not become more than slightly opalescent on the addition of silver nitrate solution.

Carbonates. — Heat 0.5 gm. of magnesium oxide with 10 cc. of water, and pour this mixture into 10 cc. of dilute acetic acid. Solution should take place without any effervescence; only a few individual gas bubbles may occur.

Barium. — The solution of 1 gm. of magnesium oxide in 10 cc. of hydrochloric acid and 10 cc. of water should not be rendered turbid on the addition of dilute sulphuric acid.

Calcium. — On shaking 1 gm. of magnesium oxide with 20 cc. of water and filtering, the filtrate, on the addition of ammonium oxalate solution, should not exhibit more than a slight opalescence within five minutes.

Heavy Metals. — The solution of 1 gm. of magnesium oxide in 20 cc. of acetic acid (sp. gr. 1.041), should not be affected by hydrogen sulphide water.

Iron. — The solution of 1 gm. of magnesium oxide in 10 cc. of hydrochloric acid and 10 cc. of water should not immediately acquire a blue color on the addition of 0.5 cc. of potassium ferrocyanide solution.

II

MAGNESIUM OXIDE, FREE FROM SULPHATES

TESTS OF PURITY

The tests as given for Magnesium Oxide should be made upon this article, and in addition the following one:

Sulphates. — Dissolve 3 gm. of magnesium oxide in 20 cc. of hydrochloric acid, dilute the solution to about 100 cc., heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form within twelve hours.

MAGNESIUM SULPHATE

$MgSO_4 + 7H_2O$. Mol. Wt. 246.53.

Clear, colorless, prismatic crystals, only very slightly efflorescent in air. Soluble in 1 part of cold, and in 0.3 part of boiling, water, but insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Chlorides. — Dissolve 1 gm. of magnesium sulphate in 20 cc. of water, and add 1 cc. of nitric acid; the solution should not be affected by silver nitrate solution.

Phosphates, Arsenates, and Heavy Metals. — The tests are to be carried out as detailed under Magnesium Chloride.

Iron. — 20 cc. of the 1:20 aqueous solution, slightly acidified with nitric acid and boiled, should not show a red color on the addition of potassium sulphocyanate solution.

Arsenic. — A mixture of 1 gm. of powdered magnesium sulphate and 3 cc. of stannous chloride solution should not acquire a dark color on standing one hour.

MAGNESIUM AND AMMONIUM CHLORIDE

$MgCl_2 \cdot NH_4Cl + 6H_2O$. Mol. Wt. 256.87.

Rhombic crystals, or white crystalline powder, easily soluble in water. Its principal use is in the preparation of magnesia mixture for the determination of phosphoric acid.

TESTS OF PURITY

Sulphates; Heavy Metals; Calcium and Barium. — The tests are to be made as detailed under Magnesium Chloride.

Phosphates and Arsenates. — Dissolve 5 gm. of magnesium and ammonium chloride in 10 cc. of water, and add 5 cc. of ammonia water. No turbidity or precipitate should form within twelve hours.

MANGANESE CHLORIDE

(Manganous Chloride)

$MnCl_2 + 4H_2O$. Mol. Wt. 197.96.

Reddish, hygroscopic crystals, easily soluble in water (about 1 : 1), and also soluble in alcohol.

TESTS OF PURITY

Sulphates. — Dissolve 1 gm. of manganous chloride in 10 cc. of water, add 0.25 cc. of hydrochloric acid, and some barium chloride solution. No change should be noticeable.

Chlorine. — The solution of 1 gm. of manganous chloride in 40 cc. of water and 5 cc. of 16 per cent sulphuric acid should not be immediately rendered blue by zinc iodide-starch solution.

Calcium. — 20 cc. of the 1 : 20 aqueous solution should not be immediately rendered turbid on the addition of 2 cc. of ammonium oxalate solution.*

* On allowing this mixture to stand for some time, manganous oxalate separates in the form of acicular crystals.

Iron, other Foreign Metals; Salts of Magnesium and the Alkalies. —

(*a*) Dissolve 1 gm. of manganous chloride in 15 cc. of water, add 1 cc. of hydrochloric acid, 3 cc. of chlorine water, and heat to boiling. The solution, when cold, should not acquire a red color on the addition of potassium sulphocyanate solution, nor should any change appear on the addition of an excess of hydrogen sulphide water.

(*b*) Precipitate the manganese from a solution of 3 gm. of manganous chloride in 50 cc. of water by adding ammonium carbonate solution (5 gm. of ammonium carbonate in 50 cc. of water). Filter, evaporate the filtrate, and ignite the residue. It should not weigh more than 0.001 gm.

Zinc. — Dissolve 1 gm. of manganous chloride and 1 gm. of sodium acetate in 10 cc. of water, and add a few drops of acetic acid and hydrogen sulphide water. No white precipitate soluble in hydrochloric acid should form.

MANGANESE DIOXIDE

(MANGANESE PER- OR SUPER-OXIDE; PYROLUSITE)

MnO_2. Mol. Wt. 87.0.

Grayish-black to steel-blue, brittle, or fibrous pieces producing a black, or grayish-black, streak. The preparation contains about 90 per cent of MnO_2.

TEST OF PURITY

Quantitative Determination. — Weigh off 1.0875 gm. of the manganese dioxide, previously very finely powdered and dried to constant weight at 100° C. Place in a flask provided with a Bunsen rubber valve, and add 75 cc. (3 pipettefuls of 25 cc. each) of a solution of 100 gm. of pure, crystallized ferrous sulphate and 100 cc. of pure, concentrated sulphuric acid in 1000 parts of water.* Close the flask with the stopper bearing the Bunsen valve, and heat until the manganese dioxide is completely decomposed, leaving no dark-colored residue. Then cool, making sure the valve is closed as indicated by the collapsing of the rubber tubing.

When cold, dilute with 200 cc. of water, and titrate with semi-normal potassium permanganate until the faint red color no longer disappears, but persists for half a minute (further decolorization is neglected). From the quantity of permanganate corresponding by calculation to the 75 cc. of ferrous sulphate solution is now deducted the quantity of permanganate actually used. Each cubic centimeter of the difference represents 0.02175 gm., or 2 per cent, of MnO_2.

MANGANESE SULPHATE

(Manganous Sulphate)

$MnSO_4 + 4H_2O$. Mol. Wt. 223.12.

Pale-red, monoclinic crystals, soluble in 0.8 part of water, and insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of manganese sulphate in 20 cc. of water should not be rendered more than slightly opalescent on the addition of 1 cc. of nitric acid, followed by silver nitrate solution.

Iron, Zinc, and other Foreign Metals; Salts of Magnesium and the Alkalies. — The tests are carried out as detailed under Manganese Chloride.

Calcium. — The solution of 1 gm. of manganese sulphate in 20 cc. of water should be perfectly clear, and should afford no immediate reaction on the addition of 2 cc. of ammonium oxalate solution.*

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* This ferrous sulphate solution is standardized against semi-normal potassium permanganate solution on the same day, using the same pipette.

MERCURY

Hg. Atomic Wt. 200.

A liquid metal with a silvery surface; completely volatilized by heat, without leaving a residue.

TESTS OF PURITY

Foreign Metals. —

(*a*) On heating 20 gm. of mercury in a porcelain dish under a good draft, no weighable residue should remain.

(*b*) On boiling 5 gm. of mercury with 5 cc. of water and 4.5 gm. of sodium thiosulphate for about one minute, in a test-tube, the mercury must not lose its luster, and should acquire at most only a slight yellowish tinge.

MERCURY BICHLORIDE

(MERCURIC CHLORIDE; CORROSIVE SUBLIMATE)

$HgCl_2$. Mol. Wt. 270.9.

White, translucent, prismatic crystals. Mercuric chloride is soluble in 16 parts of cold, and in 3 parts of boiling, water; in 3 parts of alcohol and in 12 to 14 parts of ether. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Foreign Matter not Precipitated by Hydrogen Sulphide. — Dissolve 5 gm. of mercuric chloride in 100 cc. of water, add 5 cc. of hydrochloric acid, and pass into the solution hydrogen sulphide gas until the mercury has been completely precipitated. Then filter, and evaporate the colorless filtrate to dryness on the water-bath. No weighable residue should remain.

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* See note under Manganese Chloride.

Arsenic. — Shake the mercury sulphide obtained in above test with a mixture of 5 cc. of ammonia water and 45 cc. of water, filter, and acidulate the filtrate with hydrochloric acid. Neither a yellow color nor a yellow precipitate should appear.

Mercurous Chloride and other Substances Insoluble in Ether. — 1 gm. of powdered mercuric chloride should be completely soluble in 25 cc. of ether.

MERCURY NITRATE

(MERCUROUS NITRATE)

$HgNO_3 + H_2O$. Mol. Wt. 280.05.

Colorless, monoclinic tables or prisms, soluble in about 2 parts of warm water, affording a solution with an acid reaction. On the addition of much water the solution is decomposed with the separation of a light-yellow precipitate. Solution is best effected by means of water acidified with nitric acid.

TESTS OF PURITY

Non-volatile Matter. — On igniting 2 gm. of mercurous nitrate, no weighable residue should remain.

Mercuric Salts. — Dissolve 1 gm. of mercurous nitrate in 5 cc. of water with 3 to 5 drops of nitric acid. Dilute the solution with 15 cc. of water, add an excess of hydrochloric acid, filter, and add hydrogen sulphide water to the filtrate. More than traces of a precipitate should not be visible.

MERCURY OXIDE, RED AND YELLOW

(MERCURIC OXIDE)

HgO. Mol. Wt. 216.

A heavy, reddish-yellow or yellow powder, easily soluble in dilute hydrochloric or nitric acid, giving a clear solution.

TESTS OF PURITY

Non-volatile Matter. — On igniting 2 gm. of mercuric oxide, no weighable residue should remain.

Chlorides. — The solution of 1 gm. of mercuric oxide in 5 cc. of nitric acid and 15 cc. of water should not be rendered more than slightly opalescent on adding silver nitrate solution.

Sulphates. — The solution of 1 gm. of mercuric oxide in 5 cc. of nitric acid and 15 cc. of water should show no change on the addition of barium nitrate solution.

Nitrates. — Dissolve 1 gm. of mercuric oxide in 2 cc. of water and 2 cc. of sulphuric acid, and overlay this mixture with 1 cc. of ferrous sulphate solution. No colored zone should form even on long standing.

MERCURY AND POTASSIUM IODIDE

(Mercuric Potassium Iodide)

$HgI_2 \cdot 2KI$. Mol. Wt. 785.6.

Sulphur-yellow, crystalline pieces, deliquescent in moist air.

TEST OF PURITY

Solubility. — 5 gm. of mercuric potassium iodide should completely dissolve in 10 cc. of water, and yield a clear solution which should remain clear even after adding 80 cc. of water.

METAPHENYLENEDIAMINE HYDROCHLORIDE

(Metadiaminobenzene Hydrochloride)

$C_6H_4(NH_2)_2 \cdot 2HCl$. Mol. Wt. 181.06.

A white, or slightly reddish-white, crystalline powder, easily soluble in water. Metaphenylenediamine hydrochloride is used as a reagent in the form of a 1:200 solution. Should this solution have a color, it is to be decolorized before use by heating with previously ignited animal charcoal.

METHYL ORANGE

(SODIUM SALT OF PARADIMETHYLAMINOAZOBENZENESULPHONIC ACID)

$(CH_3)_2 \cdot N \cdot C_6H_4 \cdot N : N \cdot C_6H_4 \cdot SO_3Na$. Mol. Wt. 327.24

An orange-yellow powder, easily soluble in water. As an indicator, a solution of 0.1 gm. in 100 cc. of water is used.

TEST OF SENSITIVENESS

To 100 cc. of distilled water in a Jena flask add 1 drop of the above methyl orange solution. On adding to this solution one drop of decinormal hydrochloric acid, the light-yellow color of the water should change to a red, and on the further addition of one drop of decinormal potassium hydroxide the original color should be restored.

MIXTURE, KASSNER'S

This is a mixture of barium dioxide and potassium ferricyanide, used for generating oxygen. The constituents of the mixture are kept stored in separate containers.

NITROBENZALDEHYDE, ORTHO

$C_6H_4(NO_2)CHO$. Mol. Wt. 151.07.

Light-yellow needles, melting between 45 and 46° C., and easily soluble in alcohol and in ether.

NITRON

FOR THE GRAVIMETRIC DETERMINATION OF NITRIC ACID

(1.4 DIPHENYL–3.5 ENDANILODIHYDROTRIAZOL)

$C_{20}H_{16}N_4$. Mol. Wt. 312.28.

Shining, yellow leaflets, or amorphous powder, melting at 189° C. with decomposition. It is soluble in alcohol, chloroform, acetone, and ethyl acetate, difficultly soluble in ether, and insoluble in water. In alcohol, the substance undergoes partial decomposition, indicated by the red color of the solution. Nitron is used as a reagent in the form of a 10 per cent solution in 5 per cent acetic acid, according to the method of M. Busch.

TEST OF PURITY

1 gm. of nitron should easily and almost completely dissolve in 10 cc. of 5 per cent acetic acid.

NOTE. — Literature: M. Busch, "Gravimetrische Bestimmung der Salpetersaüre," Ber., **38**, 861 (1905); J. Chem. Soc., **88**, II, 282 (1905). A. Gutbier, Ztschr. angew. Chem., **18**, 494 (1905); J. Chem. Soc., **88**, II, 418 (1905).

NITROPHENOL, ORTHO

$C_6H_4OH{\cdot}NO_2$. Mol. Wt. 139.08.

Sulphur-yellow needles or prisms, melting at 44 to 45° C., easily soluble in alcohol and in ether, freely soluble in hot water, and but slightly soluble in cold water.

NITROPHENOL, PARA

$C_6H_4OH \cdot NO_2$. Mol. Wt. 139.08.

Colorless needles or monoclinic prisms, melting at 112° C. and easily soluble in alcohol; to some extent also in water.

NITROSOBETANAPHTHOL

$C_{10}H_6(NO)(OH)$. Mol. Wt. 173.10.

Orange-brown crystals, melting at 109.5° C., insoluble in cold water, very difficultly soluble in boiling water, and very easily soluble in ether, benzene, and hot alcohol. It is used principally for the qualitative and quantitative separation of nickel and cobalt.

PALLADIUM

Pd. Atomic Wt. 106.5

Palladium occurs in the form of foil and wire which in appearance greatly resemble platinum; also in the form of palladium-sponge, a gray spongy mass, and as palladium black. The compact metal is soluble in nitrohydrochloric acid. Palladium sponge is soluble also in hydrochloric acid in the presence of air.

TESTS OF PURITY

Differentiation Between Palladium Foil and Platinum Foil. —On placing 1 drop of an alcoholic solution of iodine on palladium foil, and allowing it to evaporate spontaneously in the air, a black spot will be formed on the palladium which will disappear on heating the foil to redness. On platinum foil similarly treated, no spot is formed.

Copper and Iron. — Dissolve the palladium in nitrohydrochloric acid, and evaporate the excess of acid on the water-bath. Dissolve the residue in water, and add ammonia water until the flesh-colored precipitate of ammonium palladous chloride first formed redissolves. Then pass into the solution gaseous hydrochloric acid, whereby the palladium is precipitated as yellow palladosamine chloride, while iron and copper remain in solution. The precipitate is filtered off, and the filtrate treated with ammonia water in excess. No coloration or precipitate should ensue.

NOTE. — Regarding the quantitative determination of palladium, see Fresenius, Anleitung zur quantitativen chemischen Analyse, 6 ed., **1**, 348, 481; Fresenius-Cohn, Quantitative Chemical Analysis, **1**, 389.

PALLADIUM CHLORIDE

(PALLADOUS CHLORIDE)

$PdCl_2$. Mol. Wt. 177.4.

Dark-brown, deliquescent mass, easily soluble in water. Regarding the quantitative determination see the note under Palladium.

PALLADIUM NITRATE

(PALLADOUS NITRATE)

$Pd(NO_3)_2$. Mol. Wt. 230.58.

A brown, deliquescent salt, yielding a turbid solution with water, due to the almost invariable presence of some basic salt. Regarding the quantitative determination, see the note under Palladium.

PALLADIUM AND SODIUM CHLORIDE

(SODIUM PALLADOUS CHLORIDE)

$PdCl_2 \cdot 2NaCl$. Mol. Wt. 294.4.

A red, deliquescent salt, soluble in water and in alcohol. Regarding the quantitative determination, see the note under Palladium.

PHENACETOLIN

A brown dye, soluble in alcohol. Phenacetolin is a product resulting from the action of concentrated sulphuric acid on glacial acetic acid and phenol. The indicator solution is prepared by digesting 1 gm. of phenacetolin with warm alcohol, then diluting to 100 cc., and filtering.

TEST OF SENSITIVENESS

Add 2 drops of the indicator solution to 100 cc. of distilled water. On the addition of 0.05 cc. of decinormal potassium carbonate, the light-brown color of the water should pass into pink; on the further addition of 0.05 cc. of decinormal hydrochloric acid, the color should change to a golden yellow.

PHENOLPHTHALEIN

$C_{20}H_{14}O_4$. Mol. Wt. 318.11.

A white or yellowish-white powder, easily soluble in alcohol, and melting between 250 and 253° C. Phenolphthalein is used as an indicator in the form of a solution of 1 gm. in 100 cc. of neutral 95 per cent alcohol.

TESTS OF PURITY

Non-volatile Matter. — 0.5 gm. of phenolphthalein when ignited on platinum foil should yield no weighable residue.

Solubility in Alcohol. — 1 gm. of phenolphthalein should give a clear solution with 10 cc. of 95 per cent alcohol. The 1:100 alcoholic solution should be colorless.

Sensitiveness. — To 250 cc. of water, which has been well-boiled and then cooled, add 3 to 5 drops of phenolphthalein solution; the solution should not require more than 0.05 cc. of decinormal potassium hydroxide to effect a change from colorless to violet (not red).

Fluorane. — 0.5 gm. of phenolphthalein should completely dissolve in a mixture of 1 cc. of sodium hydroxide solution (sp. gr. 1.168) and 50 cc. of water.

PHENYLHYDRAZINE

$C_6H_5 \cdot NH \cdot NH_2$. Mol. Wt. 108.14.

Colorless or slightly yellowish, highly refractive liquid, boiling at 233° C., and solidifying when cold with the formation of monoclinic plates, melting at 23° C. Phenylhydrazine is but slightly soluble in cold water, but is more readily soluble in hot water, and is easily soluble in alcohol and in ether.

TEST OF PURITY

Solubility. — 2 cc. of phenylhydrazine, when shaken with 20 cc. of 2 per cent acetic acid, should afford a clear solution.

PHLOROGLUCIN

$C_6H_3(OH)_3 + 2H_2O$. Mol. Wt. 162.08.

A white, or slightly yellowish, crystalline powder, which loses its water of crystallization at 100° C., and which, on being rapidly heated, melts at 217 to 219° C., but which, when slowly heated, melts at a much lower temperature (200 to 209° C.). Phloroglucin is easily soluble in water, alcohol, and ether.

TEST OF PURITY

Diresorcin. — Warm a few milligrams of phloroglucin with 1 cc. of concentrated sulphuric acid and 1 to 2 cc. of acetic anhydride for five to ten minutes in a boiling water-bath. No blue color should develop.

PLATINUM

Pt. Atomic Wt. 194.8.

Platinum occurs in compact form as foil or wire, and in a state of very fine subdivision as platinum sponge and platinum black. Compact platinum has a silver-white color; platinum sponge is a grayish, spongy mass, while platinum black is a black powder.

TESTS OF PURITY

Foreign Metals. — Dissolve 1 gm. of the metal in nitrohydrochloric acid, evaporate the solution to dryness on the water-bath, and convert the residual platinum chloride into platinum sponge by strongly igniting. On now treating the sponge with nitric acid and filtering, the filtrate should not leave more than traces of residue on being evaporated.

Silver. — Dissolve platinum in nitrohydrochloric acid, evaporate the solution to dryness on the water-bath, and dissolve the residue in water. No white residue should remain.

NOTE. — Regarding the further testing of platinum for slight impurities, see the paper by F. Mylius and P. Foerster: "Uber die Herstellung und Beurteilung von reinem Platin," Ber., **25**, 665 (1892); J. Chem. Soc., **62**, 789 (1892).

PLATINUM CHLORIDE

(PLATINIC CHLORIDE; CHLORPLATINIC ACID)

$H_2PtCl_6 + 6H_2O$. Mol. Wt. 517.61.

A brownish-red, crystalline, very hygroscopic, saline mass, soluble in water, alcohol, and ether, with yellow color. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Solubility in Absolute Alcohol. — 1 gm. of platinum chloride should dissolve completely in 10 cc. of absolute alcohol, yielding a clear solution. Platinic chloride should also afford a clear, pure yellow solution with water; any red or dark-brown tinge would indicate the presence of platinous chloride or iridium.

Metals Soluble in Nitric Acid. — Strongly ignite 2 gm. of platinic chloride. A residue of platinum sponge weighing 0.752 gm. should be obtained. Digest this residue with dilute nitric acid (5 cc. of nitric acid and 20 cc. of water) on the water-bath for fifteen minutes, then filter, evaporate the filtrate on the water-bath, and ignite the residue so obtained. The weight of the latter should not exceed 0.005 gm.

Sulphates. — The solution of 1 gm. of platinic chloride in 20 cc. of water should give no precipitate of barium sulphate on adding barium chloride solution, and setting aside three hours.

Nitrates. — On mixing 2 cc. of the 1:10 solution with 2 cc. of concentrated sulphuric acid, and overlaying this mixture with 2 cc. of ferrous sulphate solution, no brownish-red zone should develop at the contact-surfaces of the two liquids, even on long standing.

Barium Salts. — The 1:20 solution of platinic chloride should afford no precipitate of barium sulphate on adding a few drops of sulphuric acid, and allowing to stand three hours.

POTASSIUM ACETATE SOLUTION

A clear, colorless liquid, neutral, or at most slightly acid to phenolphthalein; specific gravity 1.176 to 1.18. 100 parts contain about 33 parts of potassium acetate.

TESTS OF PURITY

Chlorides. — Dilute 5 cc. of potassium acetate solution with 20 cc. of water, and add 5 cc. of nitric acid followed by silver nitrate solution. At most a slight opalescent turbidity should develop.

Sulphates. — Dilute 10 cc. of potassium acetate solution with 10 cc. of water, acidify with hydrochloric acid and add barium chloride solution. No change should appear.

Heavy Metals. —

(*a*) 15 cc. of potassium acetate solution diluted with 15 cc. of water should not become colored on the addition of hydrogen sulphide water; nor should a precipitate form.

(*b*) On diluting 5 cc. of potassium acetate solution with 20 cc. of water and adding 1 cc. of hydrochloric acid, the solution should not acquire a red color with potassium sulphocyanate solution.

Calcium.—Dilute 10 cc. of potassium acetate solution with 10 cc. of water, and add ammonium oxalate solution. No precipitate of calcium oxalate should form on standing three hours.

POTASSIUM ANTIMONATE

(POTASSIUM ACID PYROANTIMONATE *)

$K_2H_2Sb_2O_7 + 4H_2O$. Mol. Wt. 504.78.

A white, granular, crystalline powder, difficultly soluble in cold water (1:250), and more readily soluble in boiling water (1:90).

TESTS OF PURITY

The aqueous solution of 1 gm. of potassium antimonate in 100 cc. of boiling water should be neutral to litmus paper.

On adding 1 gm. of potassium chloride, dissolved in 10 cc. of water, to 20 cc. of the 1:100 aqueous solution, no precipitate should form within fifteen minutes.

To 20 cc. of the 1:100 aqueous solution add 1 gm. of ammonium chloride dissolved in 10 cc. of water containing 2 or 3 drops of ammonia water. No precipitate should form within fifteen minutes.†

POTASSIUM BICARBONATE

$KHCO_3$. Mol. Wt. 100.15.

Colorless, transparent, rhombic prisms or plates, soluble in 4 parts of water.

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[*] The aqueous solution of potassium antimonate is used as a reagent for sodium. It is best to prepare the solution just before use by boiling the salt with water, and filtering. The solutions to be tested for sodium should be neutral or slightly alkaline.

[†] If a solution of 1 gm. of ammonium chloride in 10 cc. of water is added to 20 cc. of the 1:100 aqueous solution, a voluminous flocculent precipitate forms within a few minutes, if the addition of ammonia water be omitted.

TESTS OF PURITY

Sulphates. — On boiling the solution of 3 gm. of potassium bicarbonate in 50 cc. of water and 6 cc. of hydrochloric acid for several minutes, and then adding barium chloride solution, no precipitate of barium sulphate should form within twelve hours.

Chlorides. — Dissolve 3 gm. of potassium bicarbonate in 50 cc. of water, add 10 cc. of nitric acid and some silver nitrate solution. More than a slight opalescence should not develop.

Nitrates. — Dissolve 3 gm. of potassium bicarbonate in 20 cc. of water and 10 cc. of 16 per cent sulphuric acid; on adding to the solution 1 drop of a 1:1000 indigo solution and 10 cc. of concentrated sulphuric acid, the blue color should not disappear.

Silica. — Dissolve 5 gm. of potassium bicarbonate in 20 cc. of water and 15 cc. of hydrochloric acid, and evaporate the solution on the water-bath in a platinum dish. Dry the residue for half an hour at 120° C., and then dissolve it in 25 cc. of water with 3 cc. of hydrochloric acid. The solution should be perfectly clear.

Lime, Alumina, and Heavy Metals. — Dissolve 5 gm. of potassium bicarbonate in 25 cc. of water and 15 cc. of diluted acetic acid. Add 5 cc. of ammonia water and heat for half an hour on the water-bath. No flocks should separate, nor should any precipitate form. Furthermore, on adding to the slightly alkaline solution some ammonium oxalate solution and ammonium sulphide solution, no change should appear.

Phosphates. — Dissolve 5 gm. of potassium bicarbonate in 50 cc. of water, add to the solution 50 cc. of nitric acid, 25 cc. of ammonium molybdate solution, and heat at 30 to 40° C. for two hours. No yellow precipitate should form.

Iron. — Dissolve 1 gm. of potassium bicarbonate in 3 cc. of hydrochloric acid and 10 cc. of water, add a drop of nitric acid and boil, then add potassium sulphocyanate solution. No red color should develop.

Residue on Ignition. — 100 parts of potassium bicarbonate, on ignition, should leave 69 parts of residue.

Quantitative Determination. — On titrating 1 gm. of potassium bicarbonate with normal hydrochloric acid, using methyl orange as indicator, 10 cc. of acid should be required.

1 cc. of normal HCl = 0.10015 gm. of $KHCO_3$, log. 00065.

POTASSIUM BINIODATE

$KIO_3 \cdot HIO_3$. Mol. Wt. 390.09.

Small, white crystals, clearly and completely soluble in 20 parts of cold water.

TESTS OF PURITY

Quantitative Determination. — The quantitative determination may be carried out either acidimetrically or iodometrically. The salt to be used for the quantitative determination must first be brought to constant weight at 98° C. in a drying-oven.

(*a*) For the acidimetric determination about 3.5 gm. of potassium biniodate are dissolved in 200 cc. of water. The titration is made with fifth normal potassium hydroxide in the boiling solution, using phenolphthalein as indicator.

1 cc. of fifth normal KOH = 0.078018 gm. of $KIO_3 \cdot HIO_3$, log. 89218.

(*b*) The iodometric determination is made as follows: Dissolve 0.10 to 0.15 gm. of potassium biniodate in 20 cc. of water, add 3 gm. of potassium iodide and 5 cc. of hydrochloric acid, then dilute the liquid with 100 cc. of water, and titrate the liberated iodine with decinormal sodium thiosulphate.

1 cc. of decinormal $Na_2S_2O_3$ = 0.0032507 gm. of $KIO_3 \cdot HIO_3$, log. 51199.

Potassium biniodate may also be tested as to the correctness of its composition by comparing the iodine found in *b* with the quantity of iodine liberated from potassium biniodate by potassium iodide without the addition of an acid.

As will be seen from the equations given below, when potassium biniodate is decomposed by potassium iodide, without the addition of an acid, it liberates exactly one twelfth of the quantity of iodine which is liberated if the same quantity of potassium biniodate is decomposed by potassium iodide in the presence of hydrochloric or sulphuric acid.

The determination of the quantity of iodine liberated, when the decomposition of the biniodate takes place in the absence of acid, is carried out as follows: To the solution of 1 gm. of potassium biniodate in 20 cc. of water, add 3 gm. of perfectly neutral potassium iodide. Dilute with 100 cc. of water, and titrate the liberated iodine with decinormal sodium thiosulphate. It is necessary in carrying out this determination to use water perfectly free from carbon dioxide, *i.e.*, thoroughly boiled.

The iodine determined by this method, as well as that obtained under *b*, and, of course, also the number of cubic centimeters required in the titrations, provided the same quantities of potassium biniodate are used, bear the ratio 1 : 12.

This determination is of special value because the correct composition of the preparation may be controlled even without knowing the exact titer of the sodium thiosulphate solution.

NOTE. — The course of decomposition of biniodate by potassium iodide in the absence of acid may be shown by the following equations:

1. $5KHI_2O_6 + 5KI = 10KIO_3 + 5HI$;
2. $KHI_2O_6 + 5HI = KIO_3 + 6I + 3H_2O$; hence
3. $6KHI_2O_6 + 5KI = 11KIO_3 + 6I + 3H_2O$.

One KHI_2O_6, therefore, liberates one I.

In the presence of an acid, the reaction is as follows:

$KHI_2O_6 + 10KI + 11HCl = 11KCl + 6H_2O + 12I$.

Here, therefore, one KHI_2O_6 liberates twelve I.

POTASSIUM BISULPHATE

(Potassium Acid Sulphate)

$KHSO_4$. Mol. Wt. 136.21.

Colorless crystals, soluble in 2 parts of water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Heavy Metals. — The solution of 1 gm. of potassium bisulphate in 20 cc. of water should not be affected by hydrogen sulphide water; nor by ammonium sulphide solution when rendered slightly alkaline with ammonia water.

Chlorides. — 20 cc. of the 1:20 aqueous solution should not be rendered turbid on the addition of silver nitrate solution.

Arsenic. — The mixture of 1 gm. of finely powdered potassium bisulphate with 3 cc. of stannous chloride solution should not acquire a dark color within one hour.

Quantitative Determination. — Dissolve 1 gm. of potassium bisulphate in 50 cc. of water, and titrate with normal potassium hydroxide, using methyl orange as the indicator.

1 cc. of normal KOH = 0.13621 gm. of $KHSO_4$, log. 13421.

POTASSIUM BISULPHITE

(Potassium Acid Sulphite)

$KHSO_3$. Mol. Wt. 120.21.

A white, crystalline powder, having an odor of sulphurous acid and easily soluble in water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Heavy Metals and Arsenic. — Evaporate to dryness on the sand-bath a mixture of 5 gm. of potassium bisulphite and 5 cc. of sulphuric acid (sp. gr. 1.84), and dissolve the residue in 20 cc. of water. 10 cc. of this solution should show no change on the addition of hydrogen sulphide water. On adding to the other 10 cc. of the potassium sulphate solution a solution of ammonium molybdate in nitric acid, and heating the mixture to 70 to 80° C., the liquid should not acquire a yellow color, nor should a yellow precipitate form.

Quantitative Determination. — Dissolve 1 gm. of potassium bisulphite in boiled water and dilute to 100 cc. Allow the solution to run from a burette into a mixture of 30 cc. of decinormal iodine solution and 5 cc. of hydrochloric acid, with constant shaking, until decolorization is complete.

1 cc. of decinormal I = 0.0060105 gm. of $KHSO_3$, log. 77887.

POTASSIUM BITARTRATE

(Potassium Acid Tartrate)

$KHC_4H_4O_6$. Mol. Wt. 188.19.

A white, crystalline powder, soluble in 192 parts of cold, and in 20 parts of boiling, water, and insoluble in alcohol. Potassium bitartrate is also soluble in solutions of sodium hydroxide and potassium carbonate, with the evolution of carbon dioxide. The preparation contains 100 per cent of $KHC_4H_4O_6$.

TESTS OF PURITY

Moisture. — 5 gm. of potassium bitartrate, when dried at 100° C., should not suffer any loss in weight.

Chlorides. — Dissolve 1 gm. of potassium bitartrate in 20 cc. of water and add 5 cc. of nitric acid. The solution must not be rendered more than slightly opalescent on the addition of silver nitrate solution.

Sulphates. — Dissolve 1 gm. of potassium bitartrate in 20 cc. of water, add 5.0 cc. of nitric acid and barium nitrate solution. No precipitate should form within twelve hours.

Ammonium Compounds. — On heating 2 gm. of potassium bitartrate with 10 cc. of sodium hydroxide solution no vapors of ammonia should be evolved (to be ascertained by moist litmus paper).

Calcium. — Dissolve 1 gm. of potassium bitartrate in 5 cc. of diluted acetic acid and 25 cc. of water, with the aid of heat. Allow to become perfectly cold, filter, and to the filtrate add a few drops of ammonium oxalate solution. The liquid should show no turbidity within ten minutes.

Heavy Metals. — The solution of 5 gm. of potassium bitartrate in 25 cc. of water and 25 cc. of ammonia water should show no change on the addition of hydrogen sulphide water.

Quantitative Determination. —

(*a*) Dissolve 3.7638 gm. of potassium bitartrate in 200 cc. of water, by boiling, and while boiling titrate the solution with normal potassium hydroxide, using phenolphthalein as the indicator. 20 cc. of the potassium hydroxide should be required.

1 cc. of normal KOH = 0.18819 gm. of $KHC_4H_4O_6$, log. 27459.

(*b*) Cautiously incinerate 3.7638 gm. of potassium bitartrate in a platinum crucible. When cold, dissolve the contents of the crucible in water, and titrate cold with normal hydrochloric acid, using methyl orange as the indicator. 20 cc. of the hydrochloric acid should be required.

1 cc. of normal HCl = 0.18819 gm. of $KHC_4H_4O_6$, log. 27459.

POTASSIUM BROMATE

$KBrO_3$. Mol. Wt. 167.11.

Colorless crystals or crystalline powder, soluble in 15 parts of cold, or 2 parts of boiling, water. The preparation contains 100 per cent of $KBrO_3$.

TESTS OF PURITY

Potassium Bromide. — Dissolve 2 gm. of potassium bromate in 20 cc. of water and add 5 cc. of dilute sulphuric acid. The solution should not immediately acquire a yellow color.

Quantitative Determination. — For this purpose, the salt must be dried over sulphuric acid for twenty-four hours. Then dissolve 0.10 to 0.15 gm. of the dried potassium bromate in 20 cc. of water, add 3 gm. of potassium iodide, 5 cc. of hydrochloric acid, and titrate the liberated iodine with decinormal sodium thiosulphate.

1 cc. of decinormal $Na_2S_2O_3$ = 0.00278516 gm. of $KBrO_3$, log. 44485.

POTASSIUM BROMIDE

KBr. Mol. Wt. 119.11.

White, cubical, lustrous crystals, permanent in the air; soluble in 2 parts of water and in about 200 parts of alcohol.

TESTS OF PURITY

Potassium Carbonate. — The 1 : 20 aqueous solution should be neutral; it should not render a sensitive red litmus paper blue, and should not be reddened on the addition of 1 drop of phenolphthalein solution.

Potassium Bromate. — Potassium bromide which has been triturated to powder and spread on a piece of white porcelain should not immediately acquire a yellow color on adding a few drops of dilute sulphuric acid.

Heavy Metals, Potassium Sulphate, and Barium Salts. —

20 cc. portions of the 1:20 solution should show no change by hydrogen sulphide water nor by barium nitrate solution, nor by dilute sulphuric acid.

Iodides. — Add 2 or 3 drops of ferric chloride solution to 20 cc. of the 1:20 solution, then add 5 cc. of chloroform and shake. The latter should not acquire a violet color.

Excess of Potassium Chloride.* — On adding a few drops of potassium chromate solution to 10 cc. of the aqueous solution, prepared from 3 gm. of potassium bromide, dried at 100° C., and dissolved in 100 cc. of water, the solution should not require more than 25.4 cc. of decinormal silver nitrate solution to produce a permanent red color.

POTASSIUM CARBONATE

K_2CO_3. Mol. Wt. 138.3.

A white, granular, hygroscopic powder of alkaline reaction, soluble in 1 part of water, but insoluble in absolute alcohol. The preparation should contain at least 99 per cent of K_2CO_3.

TESTS OF PURITY

Heavy Metals. — 20 cc. of the 1:20 aqueous solution should show no change of color on the addition of hydrogen sulphide water. Nor should any change of color be shown upon acidulating with hydrochloric acid and then adding hydrogen sulphide water.

Chlorides. — The solution of 1 gm. of potassium carbonate in 20 cc. of water, acidified with nitric acid, should afford at most a slight opalescence on the addition of silver nitrate solution.

Sulphates. — On acidifying 20 cc. of the 1:20 aqueous solution with hydrochloric acid, then boiling for a few minutes and adding barium chloride solution, no precipitate of barium sulphate should form within twelve hours.

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* Potassium chloride is almost always present in potassium bromide, though usually in very small quantities only. The test here given allows a maximum of 1 per cent of potassium chloride.

Nitrates. — Dissolve 0.2 gm. of potassium carbonate in 2 cc. of dilute sulphuric acid, and mix the fluid with 2 cc. of concentrated suphuric acid. On now cooling the solution, and overlaying it with 1 cc. of a ferrous sulphate solution, no reddish-brown zone should form at the contact-surfaces of the two liquids.

Potassium Cyanide. — Dissolve 0.5 gm. of ferrous sulphate in 5 cc. of water, add 5 cc. of a 5 per cent potassium carbonate solution, and 1 or 2 drops of ferric chloride solution. Heat to about 60 to 70° C., and acidulate with hydrochloric acid. No green color should develop, nor should a blue precipitate form.

Sulphides, Sulphites, and Thiosulphates. — On pouring 1 cc. of the 1:20 aqueous solution into 10 cc. of decinormal silver nitrate solution, a yellowish-white precipitate forms, which should not become darker (gray to brownish to black) on being heated to 68 to 70° C.

Phosphoric Acid. — Dissolve 5 gm. of potassium carbonate in 50 cc. of water, acidulate the solution with 50 cc. of nitric acid, and add 25 cc. of ammonium molybdate solution. No yellow precipitate should form on standing two hours at about 40° C.

Silica. — Dissolve 5 gm. of potassium carbonate in a platinum dish in 20 cc. of hydrochloric acid and 20 cc. of water, and evaporate to dryness. Dry the residue for half an hour at 120° C., and then dissolve it in 3 cc. of hydrochloric acid and 25 cc. of water. The solution must be perfectly clear.

Alumina and Calcium. — Dissolve 5 gm. of the carbonate in 25 cc. of water and 25 cc. of acetic acid (sp. gr. 1.041), add 12 cc. of ammonia water, and heat for half an hour on the water-bath. No flocks nor any precipitate should form, nor should any change take place on the addition of ammonium oxalate solution to the slightly alkaline liquid.

Quantitative Determination. Dissolve 1 gm. of potassium carbonate in 50 cc. of water, add 1 drop of methyl orange solution, and titrate with normal hydrochloric acid. At least 14.3 cc. of the hydrochloric acid should be required to produce the red end-point.

1 cc. of normal HCl = 0.06915 gm. of K_2CO_3, log. 83979.

POTASSIUM CARBONATE SOLUTION

A clear, colorless, alkaline liquid, of specific gravity 1.330 to 1.334. 100 parts contain about 33 parts of potassium carbonate.

TESTS OF PURITY

The tests to be made are those given under Potassium Carbonate. But use 3 cc. of potassium carbonate solution for each gram of potassium carbonate.

POTASSIUM CHLORATE

$KClO_3$. Mol. Wt. 122.6.

Colorless, lustrous, tabular crystals, soluble in 16 parts of cold, and in 2 parts of boiling, water, and in 130 parts of 85 per cent alcohol. The salt is practically insoluble in absolute alcohol and in ether. The aqueous solution is neutral.

TESTS OF PURITY

Alkaline Earths; Chlorides. — 20 cc. of the 1:20 aqueous solution should be affected neither by ammonium oxalate solution nor by silver nitrate solution.

Heavy Metals. — The solution of 3 gm. of potassium chlorate in 30 cc. of warm water should be perfectly clear, and should remain unchanged on the addition of hydrogen sulphide water.

Nitrates. — On heating 1 gm. of potassium chlorate with 5 cc. of sodium hydroxide solution (sp. gr. 1.3), and a mixture of 0.5 gm. of zinc dust and 0.5 gm. of powdered iron, no ammonia should be evolved (to be ascertained by moist litmus paper).

Sulphates. — On adding barium chloride solution to 20 cc. of the 1:20 aqueous solution, no precipitate of barium sulphate should form on standing twelve hours.

Arsenic. — Pour 100 cc. of hydrochloric acid over 20 gm. of potassium chlorate in a capacious porcelain dish. As soon as the evolution of chlorine has slackened, evaporate the solution on the water-bath to dryness. A Marsh apparatus is set in operation, containing 20 gm. of arsenic-free, granulated zinc and dilute (1:5) sulphuric acid. The residue from the above evaporation, dissolved in 50 cc. of water, is then introducd into the generating flask of the apparatus in small portions at a time. No deposit of arsenic should be visible in the reduction tube within two hours.

POTASSIUM CHLORIDE

KCl. Mol. Wt. 74.6.

Colorless, cubical crystals, or white, crystalline powder, soluble in 3 parts of cold, but more readily soluble in boiling, water; insoluble in absolute alcohol or ether. The aqueous solution is neutral.

TESTS OF PURITY

Heavy Metals and Alkaline Earths. — The solution of 3 gm. of potassium chloride in 50 cc. of water should not be affected by ammonium oxalate solution; nor by sodium carbonate solution; nor by ammonium sulphide solution.

Sulphates. — On adding barium chloride solution to 20 cc. of the 1:20 aqueous solution, no precipitate of barium sulphate should form on standing twelve hours.

POTASSIUM CHROMATE

(YELLOW POTASSIUM CHROMATE)

K_2CrO_4. Mol. Wt. 194.4.

Yellow, rhombic crystals, permanent in the air, soluble in 2 parts of cold water, but insoluble in alcohol. The 1:20 aqueous solution is slightly alkaline to litmus paper.

TESTS OF PURITY

Free Alkali.—The solution of 0.1 gm. of potassium chromate in 25 cc. of water should not acquire a red color on the addition of a few drops of phenolphthalein solution.

Sulphates.—Dissolve 3 gm. of potassium chromate in 100 cc. of water, and add 30 cc. of hydrochloric acid, followed by barium chloride solution. No precipitate of barium sulphate should form within twelve hours.

Chlorides.—Dissolve 1 gm. of potassium chromate in 20 cc. of water, add to the solution 1 cc. of nitric acid, and heat the mixture to about 50° C. On now adding a few drops of silver nitrate solution, no turbidity or precipitate should form within five minutes.

Alumina and Alkaline Earths.—Dissolve 2 gm. of potassium chromate in 30 cc. of water, add 5 cc. of ammonium oxalate solution, and make slightly alkaline with ammonia water. No precipitate should form within twelve hours.

Quantitative Determination.—Dissolve 1 gm. of potassium chromate in water and dilute to 100 cc. Introduce 10 cc. of this solution into a glass-stoppered flask of about 400 cc. capacity, add 2 gm. of potassium iodide, 5 cc. of 16 per cent sulphuric acid, and 350 cc. of thoroughly boiled water. Titrate the liberated iodine with decinormal sodium thiosulphate, using starch solution as the indicator.

1 cc. of decinormal $Na_2S_2O_3$ = 0.00648 gm. of K_2CrO_4, log. 81158.

POTASSIUM CYANIDE

KCN. Mol. Wt. 65.19.

A white powder, easily soluble in 2 parts of water, and readily in boiling 65 per cent alcohol. The aqueous solution is alkaline to litmus paper. The salt contains 96 to 98 per cent of KCN.

TESTS OF PURITY

Sulphides. — On adding a solution of lead acetate to the solution of 1 gm. of potassium cyanide in 20 cc. of water, the resulting precipitate should have a pure white color.

Carbonates, Sulphocyanates, and Ferrocyanides. — On adding 5 cc. of hydrochloric acid to the solution of 1 gm. of potassium cyanide in 20 cc. of water, — which should be done under a hood with a good draft, — only a slight effervescence should be perceptible. On adding a drop of ferric chloride solution to the acid liquid, neither a red nor a blue color should develop.

Sulphates. — On adding to 20 cc. of the 1:20 aqueous solution 5 cc. of hydrochloric acid, and some barium chloride solution, no turbidity should develop.

Quantitative Determination. — Dissolve 1 gm. of potassium cyanide in water and dilute to 100 cc. Dilute 10 cc. of this solution with 90 cc. of water, add a granule of sodium chloride, and titrate with decinormal silver nitrate solution until a permanent, whitish turbidity appears.

1 cc. of decinormal $AgNO_3$ = 0.013038 gm. of KCN, log. 11521.

POTASSIUM DICHROMATE

(POTASSIUM BICHROMATE)

$K_2Cr_2O_7$. Mol. Wt. 294.5.

Dark, yellowish-red, triclinic prisms or plates, soluble in 10 parts of cold, or in about 1.5 parts of boiling, water, and insoluble in alcohol. The aqueous solution reddens blue litmus paper.

TESTS OF PURITY

Sulphates. Dissolve 3 gm. of potassium dichromate in 100 cc. of water, and add 30 cc. of hydrochloric acid followed by barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. Dissolve 1 gm. of potassium dichromate in 20 cc. of water, add 10 cc. of nitric acid, and heat the mixture to about 50° C. On now adding a few drops of silver nitrate solution, no turbidity should develop nor should a precipitate form, within five minutes.

Alumina and Alkaline Earths. Add ammonium oxalate solution to 2 gm. of potassium dichromate dissolved in 30 cc. of water, and make slightly alkaline with ammonia water. No precipitate should form within twelve hours.

Quantitative Determination. Dissolve 1 gm. of potassium dichromate in water, dilute to 100 cc., and introduce 10 cc. of this solution into a glass-stoppered flask of about 400 cc. capacity, together with 2 gm. of potassium iodide and 5 cc. of 16 per cent sulphuric acid. Dilute with 350 cc. of thoroughly boiled water, and titrate the liberated iodine with decinormal sodium thiosulphate, using starch solution as the indicator.

1 cc. of decinormal $Na_2S_2O_3$ = 0.0049083 gm. of $K_2Cr_2O_7$, log. 69092.

POTASSIUM FERRICYANIDE

(Red Prussiate of Potash)

$K_3Fe(CN)_6$. Mol. Wt. 329.59.

Ruby-red, shining crystals, soluble in 2.5 parts of cold, and in 1.5 parts of boiling, water.

TESTS OF PURITY

Ferrous Salt. — On first removing the superficial layers of a crystal by washing with water, and then preparing a 3 per cent aqueous solution from the washed crystals, the solution must not acquire a blue color on the addition of a few drops of a dilute ferric chloride solution.

Sulphates. — On adding 1 cc. of hydrochloric acid to 20 cc. of the 1:20 aqueous solution, and then adding barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — Deflagrate a mixture of 0.5 gm. of powdered potassium ferricyanide and 1 gm. of potassium nitrate* by introducing small quantities at a time into a porcelain crucible heated to redness. Fuse for a few minutes over the free flame, and after the melt has cooled add 20 cc. of water. Filter, to the filtrate add 0.5 gm. of potassium nitrate, evaporate to dryness, refuse in a porcelain crucible until quiescent, and allow to cool. When cold, dissolve in 20 cc. of water, add 3 cc. of nitric acid and silver nitrate solution. The liquid should show at most a slight opalescence.

Note. — Regarding the quantitative determination of potassium ferricyanide, see Mohr's Lehrb. Chem.-anal. Titriermeth., 7 ed., p. 249; Sutton, Volumet. Anal., 9 ed., p. 210.

POTASSIUM FERROCYANIDE

(Yellow Prussiate of Potash)

$K_4Fe(CN)_6 + 3H_2O$. Mol. Wt. 422.78.

Citron-yellow, tabular crystals, which are quite permanent in the air and are soluble in 4 parts of cold, and 2 parts of boiling, water; insoluble in alcohol.

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* The potassium nitrate should be tested for chloride and perchlorate. See under Potassium Nitrate.

TESTS OF PURITY

Carbonates. — On treating 1 gm. of powdered potassium ferrocyanide with dilute sulphuric acid, there should be no evolution of gas.

Sulphates. — Dissolve 1 gm. of potassium ferrocyanide in 20 cc. of water, and add 1 cc. of hydrochloric acid followed by barium chloride solution. No immediate turbidity should ensue.

Chlorides. — Deflagrate a mixture of 0.5 gm. of powdered potassium ferrocyanide and 1 gm. of potassium nitrate,* by introducing small quantities at a time into a porcelain crucible heated to redness. Treat the residue with 20 cc. of water, filter, and to the filtrate add 3 cc. of nitric acid and silver nitrate solution. The liquid should not develop more than a slight opalescence.

NOTE. — Regarding the quantitative determination of potassium ferrocyanide, see Mohr's Lehrb. Chem.-anal. Titriermeth., 7 ed., p. 245; Sutton, Volumet. Anal., 9 ed., p. 209.

POTASSIUM HYDROXIDE

(CAUSTIC POTASH; POTASSIUM HYDRATE)

KOH. Mol. Wt. 56.15.

In the laboratory, three grades of potassium hydroxide are generally used:

1. Potassium Hydroxide, Purest.
2. Potassium Hydroxide, Purified by Alcohol.
3. Potassium Hydroxide, Purified.

These three preparations differ chiefly in their content of chloride, sulphate, silica, and alumina.

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* The potassium nitrate should be tested for chloride and perchlorate. See under Potassium Nitrate.

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POTASSIUM HYDROXIDE, PUREST

White pieces showing a crystalline structure on fracture. The preparation contains 82 to 85 per cent of KOH.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of potassium hydroxide in 50 cc. of water, add 10 cc. of hydrochloric acid, heat to boiling, and then add barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — Dissolve 1 gm. of potassium hydroxide in 20 cc. of water, and add 5 cc. of nitric acid, followed by a few drops of silver nitrate solution. Not more than a slight opalescent turbidity should develop within one minute.

Nitrates. —

(*a*) Dissolve 2 gm. of potassium hydroxide in 10 cc. of water, add 10 cc. of 16 per cent sulphuric acid, 1 drop of 1 : 1000 indigo solution, a granule of sodium chloride, and 10 cc. of concentrated sulphuric acid. The blue color of the mixture should not disappear within ten minutes.

(*b*) Dissolve 25 gm. of potassium hydroxide in 100 cc. of water in a distillation flask. To the solution add 5 gm. of zinc dust and 5 gm. of powdered iron by hydrogen. Connect the flask with a condenser and a U-tube receiver containing 3 to 5 cc. of fifth normal hydrochloric acid and 10 cc. of water; allow to stand for a few hours, and then distil off about 25 cc. over a small flame. Titrate the distillate with fifth normal potassium hydroxide, using methyl orange as the indicator. At most 0.2 cc. of fifth normal hydrochloric acid should have been required to neutralize the ammonia.

Phosphoric Acid. — Dissolve 5 gm. of potassium hydroxide in 50 cc. of water and add 50 cc. of nitric acid, followed by 25 cc. of a solution of ammonium molybdate in nitric acid. This mixture should deposit no yellow precipitate on standing two hours at about 40° C.

Silica. — Dissolve 5 gm. of potassium hydroxide in 25 cc. of water in a platinum dish, add 25 cc. of hydrochloric acid, and then evaporate to dryness on the water-bath. Dry the residue for half an hour on the sand-bath at about 120° C., and then dissolve it in 10 cc. of hydrochloric acid and 90 cc. of water. Any insoluble residue should be filtered off, washed, and ignited. Its weight should not exceed 0.0005 gm.

Alumina, Calcium, and Heavy Metals. — 5 gm. of potassium hydroxide should yield a clear and colorless solution with 10 cc. of water. To this solution add 25 cc. of acetic acid (sp. gr. 1.041), followed by 10 cc. of ammonia water, dilute with 65 cc. of water, and heat the fluid on the water-bath until the odor of ammonia has entirely disappeared. Now add 2 or 3 drops of ammonia water, and allow to stand twelve hours. At the end of this time there should be no precipitate, or, at most, only an exceedingly slight, flocculent precipitate. In the latter case the precipitate is collected by filtration, washed, and ignited. Its weight should not exceed 0.0005 gm. The following tests are now carried out with the filtrate:

To 50 cc. add a few cubic centimeters of ammonium oxalate solution. No precipitate of calcium oxalate should form within two hours.

To 50 cc. add a few drops of ammonium sulphide solution. No change should appear.

Substances Insoluble in Alcohol (Potassium Carbonate and Other Potassium Salts). — 5 gm. of potassium hydroxide should completely dissolve in 25 cc. of 85 per cent alcohol on warming, giving a perfectly clear and colorless solution.

Quantitative Determination, and Determination of the Potassium Carbonate Content. — Titrate a cold solution of 1 gm. of potassium hydroxide in 100 cc. of water with normal hydrochloric acid, using phenolphthalein as the indicator. At least 14.8 cc. of the acid should be necessary to destroy the red color. (First titration.) Now add 1 drop of methyl orange, and titrate further until the color again changes to red. In this second titration, not more than 0.15 cc. of the acid should be required (2.07 per cent K_2CO_3).*

1 cc. of normal HCl = 0.05615 gm. of KOH, log. 74935.

1 cc. of normal HCl = 0.06915 gm. of K_2CO_3, log. 83979.

II

POTASSIUM HYDROXIDE, PURIFIED BY ALCOHOL

White, very hygroscopic sticks or pieces, exhibiting a crystalline fracture. The preparation contains at least 80 per cent of KOH.

TESTS OF PURITY

Sulphates and Nitrates. — The test is carried out as detailed under Potassium Hydroxide, Purest.

Chlorides. — Dissolve 1 gm. of potassium hydroxide in 20 cc. of water, add 5 cc. of nitric acid and a few drops of silver nitrate solution. An opalescence may develop, but no precipitate should form.

Silica. — Dissolve 5 gm. of potassium hydroxide in 25 cc. of water and 25 cc. of hydrochloric acid in a platinum dish, and evaporate to dryness on the water-bath. Dry the residue for half an hour on the sand-bath at about 120° C., then treat it with 10 cc. of hydrochloric acid and 90 cc. of water, filter, wash, and ignite any undissolved residue. The weight of the latter should not exceed 0.0025 gm.

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* From the results of both titrations, the content of potassium hydroxide and potassium carbonate are calculated as follows:

The result of the second titration multiplied by two gives the number of cubic centimeters which are necessary to neutralize the potassium carbonate originally present.

On subtracting the number of cubic centimeters required in the second titration from the number of cubic centimeters used in the first titration, the remainder gives the number of cubic centimeters which were required to neutralize the potassium hydroxide.

Alumina, Calcium, and Heavy Metals. — 5 gm. of potassium hydroxide should completely dissolve in 20 cc. of water, affording a clear and colorless solution. Dilute the solution to 100 cc., and add 25 cc. of acetic acid (sp. gr. 1.041) followed by 10 cc. of ammonia water. At most a slight turbidity, but no flocculent precipitate of aluminum hydroxide should develop within five minutes; and no immediate change should appear on the addition of ammonium oxalate and ammonium sulphide solutions.

Quantitative Determination and Determination of the Potassium Carbonate Content. — Dissolve 1 gm. of potassium hydroxide in 100 cc. of water, and titrate the cold solution with normal hydrochloric acid, using phenolphthalein as the indicator. At least 14.5 cc. of the acid should be necessary to destroy the red color. Now add 1 drop of methyl orange, and titrate further until the color again changes to red. In this second titration not more than 0.25 cc. of the acid should be required (3.45 per cent K_2CO_3).*

III

POTASSIUM HYDROXIDE, PURIFIED

White, very hygroscopic sticks or pieces which exhibit a crystalline fracture. The preparation should contain at least 80 per cent of KOH.

TESTS OF PURITY

Nitrates. — Dissolve 2 gm. of potassium hydroxide in 10 cc. of water, and add 10 cc. of dilute sulphuric acid, followed by

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* See the note under Potassium Hydroxide, Purest.

1 drop of 1:1000 indigo solution, a granule of sodium chloride, and 10 cc. of concentrated sulphuric acid. The blue color of the mixture should not disappear within ten minutes.

Alumina, Calcium, and Heavy Metals. — 2.5 gm. of potassium hydroxide should completely dissolve in 10 cc. of water yielding a clear and colorless solution. Dilute the solution to 100 cc. and add 15 cc. of acetic acid (sp. gr. 1.041), followed by 10 cc. of ammonia water; a slight turbidity may form within five minutes, but no flocculent precipitate of aluminum hydroxide should develop. The solution so tested, filtered if necessary, should not exhibit an immediate turbidity on adding ammonium oxalate solution; and on the addition of ammonium sulphide solution should acquire at most a slight green color.

Quantitative Determination and Determination of the Potassium Carbonate Content. — The determinations are made as detailed under Potassium Hydroxide, Purified by Alcohol. The content of potassium carbonate should not exceed 5 per cent.

POTASSIUM HYDROXIDE SOLUTIONS

A clear, colorless liquid, of specific gravity 1.3, and containing about 32 per cent of potassium hydroxide.

Also a similar solution having a specific gravity 1.138 to 1.140 and containing about 15 per cent of potassium hydroxide.

TESTS OF PURITY

The tests and the quantitative determination, as given under Potassium Hydroxide, Purified by Alcohol, are to be made on each solution. But, of the solution having sp. gr. 1.3, use 2 cc. (2.6 gm.) in place of each gram of the solid hydroxide referred to. And, of the solution having sp. gr. 1.138 to 1.140, use 5 cc. (5.7 gm.).

POTASSIUM IODATE

KIO_3. Mol. Wt. 214.12.

A white, crystalline powder, soluble in 13 parts of cold, and in 3 parts of boiling, water. The aqueous solution should be clear and have no acid reaction. The preparation contains 100 per cent of KIO_3.

TESTS OF PURITY

Free Acid. — Dissolve about 0.5 gm. of potassium iodate in about 20 cc. of boiled water, and add a small crystal of neutral potassium iodide and a few drops of freshly prepared starch solution. The liquid should not immediately acquire a blue color.

Iodides. — Dissolve 2 gm. of potassium iodate in 20 cc. of water, add 5 cc. of dilute sulphuric acid and a few drops of starch solution. The liquid should not immediately acquire a blue color.

Quantitative Determination. — Previous to the quantitative determination, the salt should be dried for twenty-four hours over sulphuric acid.

Dissolve 0.10 to 0.15 gm. of dried potassium iodate in 20 cc. of water, add 3 gm. of potassium iodide and 5 cc. of hydrochloric acid. Dilute with 50 cc. of water, and titrate the liberated iodine with decinormal sodium thiosulphate.

1 cc. of decinormal $Na_2S_2O_3$ = 0.0035686 gm. of KIO_3, log. 55250.

POTASSIUM IODIDE

KI. Mol. Wt. 166.12.

White, cubical crystals, not becoming moist on contact with air. Potassium iodide is soluble in about 0.75 part of water, in about 12 parts of 85 per cent alcohol, and in 40 parts of absolute alcohol.

TESTS OF PURITY

Potassium Carbonate. — Potassium iodide when crushed and placed upon moistened red litmus paper should not immediately color the latter violet-blue.

On adding to a solution of 0.5 gm. of potassium iodide in 10 cc. of water 1 drop of phenolphthalein solution, no red color should develop.

Metals and Sulphates. — 20 cc. portions of the 1:20 aqueous solution should afford no reaction with either hydrogen sulphide water or barium chloride solution.

Cyanides. — On adding to 20 cc. of the 1:20 aqueous solution a granule of ferrous sulphate, 1 drop of ferric chloride solution, and 5 cc. of sodium hydroxide solution (sp. gr. 1.3), then heating the mixture to 50 to 60° C., and adding 10 cc. of hydrochloric acid, no blue color should develop.

Iodates. — 20 cc. of the 1:20 solution, freshly prepared with recently boiled and cooled water, should not immediately acquire a blue color on the addition of starch solution and 2 to 3 drops of dilute sulphuric acid.

Nitrates. — On heating 1 gm. of potassium iodide with 5 cc. of sodium hydroxide solution and a mixture of 0.5 gm. of zinc dust and 0.5 gm. of powdered iron, no vapors of ammonia should be evolved (to be ascertained by moist litmus paper).

Chlorides, Bromides, and Thiosulphates. — Dissolve 0.2 gm. of potassium iodide in 2 cc. of ammonia water, add 13 cc. of decinormal silver nitrate solution, shake, and filter. On supersaturating the filtrate with nitric acid, the solution should not become so cloudy as to be opaque nor should a dark color develop, within ten minutes.

POTASSIUM IODIDE, NEUTRAL

TESTS OF PURITY

Neutrality. — Dissolve 10 gm. of the neutral potassium iodide in 50 cc. of water, in a stoppered, flint-glass bottle, overlay with 30 cc. of ether, and add 3 drops of iodeosin solution. After vigorously shaking, the aqueous layer will exhibit a pale-red color which disappears on adding 1 drop of decinormal hydrochloric acid and thoroughly shaking.

Should the water which has been used for making the solution of potassium iodide have an alkaline reaction to iodeosin, it must be first rendered neutral to it.

Further Tests. — The tests as given under Potassium Iodide are also to be made, and in the manner there described.

POTASSIUM NITRATE

KNO_3. Mol. Wt. 101.19.

Colorless, transparent, prismatic crystals, or crystalline powder, permanent in air, and soluble in 4 parts of cold, and in less than 0.5 part of boiling, water; almost insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of potassium nitrate in 60 cc. of water, and add 0.5 cc. of hydrochloric acid followed by barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — Dissolve 1 gm. of potassium nitrate in 20 cc. of water, acidulate with 1 cc. of nitric acid and add silver nitrate solution. No turbidity should develop.

Chlorates and Perchlorates. — Gently ignite 1 gm. of potassium nitrate, dissolve the melt in 20 cc. of water, add 1 cc. of nitric acid and some silver nitrate solution. No change should appear.

Calcium and Heavy Metals. —

(*a*) The solution of 3 gm. of potassium nitrate in 50 cc. of water should not be affected by hydrogen sulphide water.

(*b*) The solution of 3 gm. of potassium nitrate in 50 cc. of water should not be affected by ammonia water; nor by the further addition of ammonium oxalate solution and of ammonium sulphide solution.

Iron. — 20 cc. of the 1:20 aqueous solution, acidulated with 1 cc. of hydrochloric acid (sp. gr. 1.124), should not be reddened by potassium sulphocyanate solution.

Nitrites. — Dissolve 1 gm. of potassium nitrate in 20 cc. of water, acidulate with 1 cc. of 16 per cent sulphuric acid, and add 1 cc. of a freshly prepared, colorless 1:200 solution of metaphenylenediamine hydrochloride.* No yellow or yellowish-brown color should develop.

POTASSIUM NITRITE

KNO_2. Mol. Wt. 85.19.

White, or slightly yellowish, tough sticks, which are deliquescent in the air, and are easily soluble in water. The aqueous solution is alkaline to litmus paper. The preparation contains at least 90 per cent of KNO_2.

TESTS OF PURITY

Heavy Metals. — On adding a few drops of ammonium sulphide solution to 20 cc. of the 1:20 aqueous solution, no change should appear.

Sulphates. — On adding to 20 cc. of the 1:20 aqueous solution 5 cc. of nitric acid and barium nitrate solution, no turbidity should develop.

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* Should the solution of metaphenylenediamine hydrochloride have a color, it is to be decolorized before using by warming with previously ignited animal charcoal.

Quantitative Determination. — Dissolve 10 gm. of potassium nitrite in water and dilute to 1000 cc. Make a mixture of 50 cc. of decinormal potassium permanganate (accurately measured) with 300 cc. of water and 25 cc. of 16 per cent sulphuric acid, warm to 40 to 50° C, and, while constantly shaking, allow the nitrite solution to run slowly into it, until the red color disappears. Care must be taken towards the end to run in the nitrite solution very slowly, because the change from red to colorless requires some time. Not more than 23.7 cc. of the potassium nitrite solution should be required.

1 cc. of decinormal $KMnO_4$ = 0.0042595 gm. of KNO_2, log. 62936.

POTASSIUM OXALATE, NEUTRAL

$K_2C_2O_4 + H_2O$. Mol. Wt. 184.308.

Rhombic prisms, soluble in 3 parts of water. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — Boil a solution of 5 gm. of potassium oxalate in 200 cc. of water, and add 5 cc. of hydrochloric acid followed by barium chloride solution. No precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — Dissolve 1 gm. of potassium oxalate in 25 cc. of water, and add 10 cc. of nitric acid and a few drops of silver nitrate solution. No turbidity should develop on shaking.

Heavy Metals. — To a solution of 1 gm. of potassium oxalate in 25 cc. of water, add hydrogen sulphide water. The solution should remain unchanged. On now adding to the liquid 5 cc. of ammonia water, no green color should develop, nor should a precipitate form.

Quantitative Determination. — Dissolve about 3.2 gm. of potassium oxalate in water and dilute to 500 cc. To 50 cc. of this solution add 6 to 8 cc. of concentrated sulphuric acid, heat the liquid to about 60° C., and titrate with decinormal potassium permanganate.

1 cc. of decinormal $KMnO_4$ = 0.0092158 gm. of $K_2C_2O_4 + H_2O$, log. 96453.

POTASSIUM PERCHLORATE

$KClO_4$. Mol. Wt. 138.6.

Colorless, rhombic prisms, soluble in about 65 parts of cold, and in 8 parts of boiling, water, and insoluble in alcohol.*

TESTS OF PURITY

Calcium; Chlorides. — 20 cc. of the 1:20 aqueous solution prepared by the aid of heat should not be affected by ammonium oxalate solution; and it should develop at most a slight opalescence on the addition of silver nitrate solution.

Heavy Metals. — 20 cc. of the 1:20 aqueous solution prepared with the aid of heat should not be affected by hydrogen sulphide water.

Nitrates. — On heating a mixture of 1 gm. of potassium perchlorate with 5 cc. of sodium hydroxide solution (sp. gr. 1.3) and a mixture of 0.5 gm. of zinc dust and 0.5 gm. of powdered iron, no vapors of ammonia should be evolved (to be ascertained by moist litmus paper).

Sulphates. — On adding barium chloride solution to 20 cc. of the 1:20 aqueous solution prepared by the aid of heat, no change should appear.

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* Potassium perchlorate is distinguished from potassium chlorate by the fact that it dissolves without color in concentrated sulphuric acid, and is not decomposed by hydrochloric acid with the liberation of free chlorine.

POTASSIUM PERMANGANATE

$KMnO_4$. Mol. Wt. 158.15.

I

POTASSIUM PERMANGANATE

Dark-violet, almost black prisms, exhibiting a steel-blue luster, soluble in 16 parts of cold, and in 3 parts of boiling, water. The 1:1000 aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates and Chlorides. — Boil 0.5 gm. of potassium permanganate with a mixture of 2 cc. of 95 per cent alcohol and 25 cc. of water, and filter. The filtrate should be colorless, and, after adding to it 2 cc. of nitric acid, barium nitrate, and silver nitrate solutions, it should not develop more than a slight opalescence.

Nitrates. — Add gradually 1 gm. of crystallized oxalic acid to a solution of 0.5 gm. of potassium permanganate in 5 cc. of water, heat to 50 to 60° C., and filter. On overlaying 1 cc. of ferrous sulphate solution on a mixture of 2 cc. of the clear colorless filtrate and 2 cc. of concentrated sulphuric acid, no dark-colored zone should form at the contact-surfaces of the two liquids.

Chlorates. — Heat 2 gm. of potassium permanganate in a platinum crucible, and gradually add small pieces of paraffin until no more glowing is observed. When cold, the residue is treated with 20 cc. of water, filtered, and 5 cc. of nitric acid and silver nitrate solution are added to the filtrate. The liquid should develop at most a slight opalescence.

Quantitative Determination. — Dissolve 3.164 gm. of potassium permanganate in water and dilute to 1000 cc. Also dissolve 6.715 gm. of sodium oxalate,* dried at 240° C. (see Sodium Oxalate, Sörensen's), in water and dilute to 1 liter (decinormal sodium oxalate). Measure off 30 cc. of the oxalate solution with a burette, add to it 6 to 8 cc. of concentrated sulphuric acid, heat to about 60° C. ,and run in the potassium permanganate solution until a permanent red color appears.

1 cc. of decinormal $Na_2C_2O_4$ = 0.003164 gm. of $KMnO_4$, log. 50024.

II

POTASSIUM PERMANGANATE, FREE FROM SULPHATES

The preparation contains 99.7 to 100 per cent of $KMnO_4$.

TESTS OF PURITY

Sulphates. — The solution of 3 gm. of potassium permanganate in 150 cc. of water with 15 cc. of 95 per cent alcohol (or a little hydrogen peroxide, 30 per cent), heated, if necessary, until perfectly decolorized, should yield a filtrate which, on the addition of 2 cc. of hydrochloric acid and barium chloride solution, should afford no precipitate of barium sulphate on standing twelve hours.

Further Tests. — The other tests and the quantitative determination as given under Potassium Permanganate are also to be made.

POTASSIUM STANNOSULPHATE

(Marignac's Salt)

$K_2Sn(SO_4)_2$. Mol. Wt. 389.42

White crystals, soluble in dilute potassium or sodium hydroxide solution, and in hydrochloric acid.

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* The proper composition of the sodium oxalate is verified and controlled by qualitative tests and by titration with fifth normal hydrochloric acid.

TEST OF PURITY

Quantitative Determination.—Dissolve 1 gm. of potassium stannosulphate in a solution of 5 gm. of sodium bicarbonate and 5 gm. of potassium and sodium tartrate in 100 cc. of water, and titrate the clear liquid with decinormal iodine, using starch solution as the indicator.

1 cc. of decinormal I = 0.019471 gm. of $K_2Sn(SO_4)_2$, log. 28939.

POTASSIUM SULPHATE

K_2SO_4. Mol. Wt. 174.36.

White, hard crystals, soluble in 10 parts of cold, and in 4 parts of boiling, water, but insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Chlorides, Heavy Metals (Fe, Cu), Calcium, and Magnesium). 20 cc. portions of the 1:20 aqueous solution should not be affected by hydrogen sulphide water, nor by ammonium oxalate, silver nitrate, and sodium phosphate solutions.

Iron.—The solution of 1 gm. of potassium sulphate in 20 cc. of water boiled with a few drops of nitric acid should remain colorless on the addition of potassium sulphocyanate solution.

POTASSIUM SULPHIDE

Leather-brown or yellowish-green pieces, which deliquesce in moist air, dissolve in 2 parts of water with but a slight residue, and afford alkaline, yellowish-green solutions. On adding acetic acid to the aqueous solution, hydrogen sulphide gas is generated in copious quantities.

POTASSIUM SULPHIDE SOLUTION

The solution contains 5 per cent of K_2S, and is intended for use in determining nitrogen according to Kjeldahl.

TEST OF PURITY

Nitrogen. — Mix 100 cc. of the potassium sulphide solution in a distilling flask with 50 cc. of nitrogen-free sodium hydroxide solution (sp. gr. 1.3). Add 1 gm. of zinc dust, and distil off about 50 cc., collecting the distillate in a U-tube receiver containing about 20 cc. of water and 2 to 3 cc. of decinormal hydrochloric acid. Titrate the distillate with decinormal potassium hydroxide, using methyl orange as the indicator. The ammonia distilled over should not have neutralized more than 0.2 cc. of the decinormal acid.

POTASSIUM SULPHOCYANATE

(Potassium Thiocyanate)

KSCN. Mol. Wt. 97.25.

Colorless, prismatic crystals, deliquescent in the air, and easily soluble in water and in alcohol.

TESTS OF PURITY

Substances Insoluble in Alcohol. — 1 gm. of potassium sulphocyanate should completely dissolve in 10 cc. of boiling absolute alcohol, and yield a clear solution.

Sulphates. — On adding to a solution of 1 gm. of potassium sulphocyanate in 20 cc. of water, a few drops of hydrochloric acid and some barium chloride solution, no turbidity should be observed within five minutes.

Heavy Metals. — On dissolving 1 gm. of potassium sulphocyanate in 20 cc. of water, and adding to the solution a few drops of ammonium sulphide solution, no precipitate should form, nor should a brown or green color develop.

Iron. — The solution of 1 gm. of potassium sulphocyanate in 20 cc. of water should remain perfectly colorless on the addition of 0.5 cc. of hydrochloric acid.

POTASSIUM SULPHYDRATE

(POTASSIUM HYDROSULPHIDE)

$2KSH + H_2O$. Mol. Wt. 162.45.

Colorless, deliquescent crystals* easily soluble in water and in alcohol. The solution is strongly alkaline to litmus paper. On boiling, the aqueous solution is decomposed with the evolution of hydrogen sulphide gas and the formation of potassium hydroxide.

TEST OF PURITY

Polysulphides. The solution of 1 gm. of potassium sulphydrate in 20 cc. of water should be colorless and clear. On acidulating the solution with hydrochloric acid, hydrogen sulphide gas is generated in copious quantities, and the fluid exhibits a slight opalescent turbidity. No separation of sulphur should occur.

POTASSIUM TETRAOXALATE

$KHC_2O_4 + H_2C_2O_4 + 2H_2O$. Mol. Wt. 254.20.

Colorless, triclinic crystals, soluble in 55 parts of water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

The tests for chlorides, sulphates, and heavy metals are to be made as detailed under Potassium Oxalate, Neutral.

Quantitative Determination.

(a) Acidimetric: Dissolve about 3.10 gm. of potassium tetraoxalate in water and dilute to 500 cc. Titrate 50 cc. of this solution, while boiling, with fifth normal potassium hydroxide, using phenolphthalein as the indicator.

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* When exposed to the air, the preparation rapidly acquires a yellow color by taking up oxygen with the formation of polysulphides. It should, therefore, be preserved in well-stoppered bottles.

1 cc. of fifth normal KOH = 0.016947 gm. of $KHC_2O_4 + H_2C_2O_4 + 2H_2O$, log. 22909.

(*b*) Oxidimetric: To 50 cc. of the solution made for test *a*, add 6 to 8 cc. of concentrated sulphuric acid, heat the liquid to about 60° C., and titrate with decinormal potassium permanganate.

1 cc. of decinormal $KMnO_4$ = 0.006355 gm. of $KHC_2O_4 + H_2C_2O_4 + 2H_2O$, log. 80312.

POTASSIUM AND SODIUM TARTRATE

(ROCHELLE SALT; SEIGNETTE SALT)

$KNaC_4H_4O_6 + 4H_2O$. Mol. Wt. 282.29.

Colorless, prismatic crystals, soluble in 1.4 parts of water, and yielding a neutral solution.

TESTS OF PURITY

Calcium. — On dissolving 1 gm. of potassium and sodium tartrate in 10 cc. of water, and adding to the solution 5 cc. of dilute acetic acid, and then shaking for a few minutes, a crystalline precipitate forms. On filtering off the latter, diluting the filtrate with an equal volume of water, and then adding 8 to 10 drops of ammonium oxalate solution, no turbidity should develop within one minute.

Heavy Metals. — The 1:20 aqueous solution should show no change with hydrogen sulphide water.

Chlorides and Sulphates. — The solution of 1 gm. of potassium and sodium tartrate in 10 cc. of water, with 2 cc. of nitric acid added, should not be affected by silver nitrate and barium nitrate solutions.

Ammonium Compounds. — On heating the solution of 1 gm. of potassium and sodium tartrate in 10 cc. of water with 10 cc. of sodium hydroxide solution (sp. gr. 1.3), no vapors of ammonia should be given off (to be ascertained by means of moist litmus paper).

PYROGALLOL

(ACID PYROGALLIC)

$C_6H_3(OH)_3$. Mol. Wt. 126.04.

White, shining needles or scales, melting at 131° C. Pyrogallol is soluble in 1.7 parts of water, in 1 part of alcohol, and in 1.2 parts of ether; it is difficultly soluble in benzene, chloroform, and carbon disulphide. The aqueous solution is slightly acid to litmus paper.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of pyrogallol, on being heated, should volatilize and leave no weighable residue.

Gallic Acid. — 2 gm. of pyrogallol must completely dissolve in 5 cc. of ether (sp. gr. 0.72), and afford a clear solution.

RESORCINOL

(RESORCIN)

$C_6H_4(OH)_2$. Mol. Wt. 110.04.

Colorless crystals, having a scarcely perceptible but peculiar odor, soluble in about 1 part of water, or alcohol, easily soluble in ether and in glycerin, difficultly soluble in chloroform and in carbon disulphide. Resorcinol volatilizes on being heated, and melts at 110 to 111° C. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Non-volatile Matter. — 1 gm. of resorcinol, on being heated and volatilized, should leave no weighable residue.

Diresorcin and Phenol. — 1 gm. of resorcinol should yield a perfectly clear solution with 20 cc. of water; and the solution on being warmed should not have a phenolic odor.

Free Acids (e.g., **Salicylic Acid**). — On dissolving 1 gm. of resorcinol in 10 cc. of alcohol, and adding 1 drop of lacmoid solution, a wine-red liquid is obtained, which, on the addition of 1 drop of decinormal potassium hydroxide, should assume a blue color.

SILVER

Ag. Atom. Wt. 107.93.

A white, shining metal in the form of foil, insoluble in hydrochloric and in cold dilute sulphuric acids; soluble in nitric and in hot concentrated sulphuric acids.

TESTS OF PURITY

Foreign Metals. — Dissolve 2 gm. of silver in the smallest possible quantity of nitric acid (sp. gr. 1.2). The solution should be colorless and there should be no insoluble residue (Sb and Sn). Dilute with about 200 cc. of water (no turbidity should occur after standing one hour — Bi), and precipitate the silver by adding hydrochloric acid to the boiling solution. Allow the precipitate to settle in a dark place, filter, and evaporate the filtrate. No weighable residue should remain.

Quantitative Determination. — This may be carried out either gravimetrically by precipitating the silver by means of hydrochloric acid from a boiling solution slightly acid with nitric acid; or the determination may be made volumetrically according to Gay-Lussac's method, which is used in all the laboratories of the German mints.

NOTE. — Regarding the manner in which this method is carried out, see Lunge, Chem.-tech. Untersuch.-Meth., 5 ed., **2**, 135; Sutton, Volumet. Anal., 9 ed., p. 303.

SILVER NITRATE

$AgNO_3$. Mol. Wt. 169.97.

Colorless, lustrous crystals or sticks, exhibiting a stellate, crystalline fracture, and yielding a clear, colorless solution with 0.6 part of water, and with about 10 parts of alcohol. The aqueous solution should be neutral to litmus paper.

TESTS OF PURITY

Chlorides. — Dissolve 5 gm. of silver nitrate in 5 cc. of water, and allow the solution to run into 100 cc. of water. No turbidity or opalescence should occur.

Potassium Nitrate. — Dissolve 0.5 gm. of silver nitrate in 0.5 cc. of water, mix the solution with 20 cc. of absolute alcohol, and shake for a few minutes. No turbidity or precipitate should form.

Salts of Copper, Bismuth, and Lead. — Dissolve 1 gm. of silver nitrate in 5 cc. of water, and add to the solution 10 cc. of ammonia water. The liquid should remain clear and colorless.

Substances not Precipitated by Hydrochloric Acid. — Dissolve 2 gm. of silver nitrate in 50 cc. of water, heat the solution to boiling, and add 3 cc. of hydrochloric acid. After the precipitate has settled, filter, and evaporate the filtrate to dryness. No weighable residue should remain.

Quantitative Determination. — This is carried out as detailed under Silver.

SILVER NITRITE

$AgNO_2$. Mol. Wt. 153.97.

Small, yellowish, acicular crystals, soluble in about 300 parts of cold water. Silver nitrite is more readily soluble in hot water, but suffers partial decomposition therein.

TESTS OF PURITY

Substances not Precipitated by Hydrochloric Acid. — Dissolve 2 gm. of silver nitrite, with heat, in 100 cc. of water, with the aid of 2 cc. of nitric acid, heat the solution to boiling, and add 3 cc. of hydrochloric acid. After the precipitate has settled, filter, and evaporate the filtrate to dryness. No weighable residue should remain.

Quantitative Determination.* — Dissolve 1.5 gm. of silver nitrite in water and dilute to 500 cc. Make a mixture containing 18 to 19 cc. of decinormal potassium permanganate solution, 20 cc. of dilute sulphuric acid, and 300 cc. of water, heat it to 40 to 50° C., and run the nitrite solution into it until the pink color just disappears. Care must be taken towards the end to introduce the nitrite solution very slowly, because the change from pink to colorless always requires some time.

1 cc. of decinormal $KMnO_4$ = 0.0076985 gm. of $AgNO_2$, log. 88640.

SODIUM

Na. Atomic Wt. 23.05.

The metal, when freshly cut, has a silver-white surface, which rapidly becomes dull on exposure to air and becomes covered with crusts of sodium oxide, sodium hydroxide, and sodium carbonate. At ordinary temperatures, sodium has the consistency of wax, but at low temperatures it is brittle.

TESTS OF PURITY

Foreign Metals. —

(*a*) 1 gm. of sodium is freed from adhering petroleum by wiping with pieces of filtering paper, then cut into small pieces, and thrown upon 20 cc. of cold water. The solution of sodium hydroxide so obtained should appear unchanged upon adding ammonium sulphide solution.

(*b*) The solution perpared from 1 gm. of sodium and 20 cc. of water should be unchanged in appearance on the addition of 10 cc. of hydrochloric acid, followed by hydrogen sulphide water.

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* Compare other methods of determining nitrites; Sutton, Volumet. Anal., 9 ed., p. 270 (1904).

SODIUM ACETATE

$NaC_2H_3O_2 + 3H_2O$. Mol. Wt. 136.12.

Colorless, transparent crystals, efflorescent in warm air, soluble in 1 part of water, in 23 parts of cold, and 1 part of boiling, alcohol.

The solution of 1 gm. of sodium acetate in 1 cc. of water is alkaline to litmus paper; it should, however, not be reddened, or only very slightly, by phenolphthalein solution.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of sodium acetate in 20 cc. of water should appear unchanged on adding 1 cc. of nitric acid followed by silver nitrate solution.

Sulphates. — 20 cc. of the 1:20 aqueous solution should not be rendered turbid by barium chloride solution.

Heavy Metals and Calcium. — 20 cc. of the 1:20 aqueous solution should not be affected by hydrogen sulphide water, nor by ammonium oxalate solution.

Iron. — The solution of 1 gm. of sodium acetate in 20 cc. of water should not be reddened on adding 1 cc. of hydrochloric acid, followed by potassium sulphocyanate solution.

SODIUM AMALGAM

Gray pieces, the size of peas, containing 2 per cent of metallic sodium.

TEST OF STRENGTH

Quantitative Determination of Sodium. — Introduce 10 gm. of sodium amalgam into 100 cc. of water, and allow the mixture to stand with repeated shaking until the evolution of hydrogen entirely ceases. Then titrate with normal hydrochloric acid, using methyl orange as the indicator.

1 cc. of normal HCl = 0.02305 gm. of Na, log. 36267.

SODIUM BICARBONATE

$NaHCO_3$. Mol. Wt. 84.05.

White, crystalline crusts, or crystalline powder, soluble in 12 parts of water, but insoluble in alcohol. The aqueous solution is faintly alkaline to litmus paper.

TESTS OF PURITY

Sulphates. — Dissolve 2 gm. of sodium bicarbonate in 30 cc. of water, add 10 cc. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form within twelve hours.

Silica. — Dissolve 5 gm. of sodium bicarbonate in 15 cc. of water and 25 cc. of hydrochloric acid in a platinum dish. Evaporate the solution on the water-bath, dry the residue for half an hour at 120° C., and then dissolve it in 3 cc. of hydrochloric acid and 25 cc. of water. The solution should be perfectly clear.

Chlorides, Thiosulphates, and Arsenic. — On adding to the solution of 1 gm. of sodium bicarbonate in 50 cc. of water 5 cc. of dilute acetic acid, followed by silver nitrate solution, at most a faint opalescence should develop.

Phosphates. — Dissolve 2 gm. of sodium bicarbonate in 20 cc. of water, add 20 cc. of nitric acid and 10 cc. of ammonium molybdate solution. On heating to 30 to 40° C., no yellow precipitate should form within two hours.

Heavy Metals. —

(*a*) The solution of 3 gm. of sodium bicarbonate in 40 cc. of water and 8 cc. of hydrochloric acid should remain unchanged on the addition of hydrogen sulphide water; on adding to the liquid 5 cc. of ammonia water and a few drops of ammonium sulphide solution, no precipitate should form, nor should a green or brown color develop.

(*b*) The solution of 1 gm. of sodium bicarbonate in 15 cc. of water and 2 cc. of hydrochloric acid should not be reddened on the addition of potassium sulphocyanate solution.

Potassium. — The yellow color imparted by sodium bicarbonate to the flame, when observed through cobalt glass, should have at most a transient violet tinge.

Monocarbonate (Neutral Sodium Carbonate). —

(*a*) A solution of 1 gm. of sodium bicarbonate in 20 cc. of water, prepared at a temperature not above 15° C., without excessive shaking, should not be immediately reddened on the addition of 3 drops of phenolphthalein solution; at all events, any slight redness that may develop should disappear on the addition of 0.2 cc. of normal hydrochloric acid.

(*b*) 1 gm. of the sodium bicarbonate dried over sulphuric acid, and ignited, should leave a residue weighing not more than 0.638 gm.

Ammonia. — On heating 1 gm. of sodium bicarbonate in a test-tube, no vapors of ammonia should be evolved (to be ascertained by moistened turmeric paper).

Sulphocyanates. — The solution of 1 gm. of sodium bicarbonate in 3 cc. of nitric acid and 47 cc. of water should not be reddened on the addition of 1 drop of ferric chloride solution.

Quantitative Determination. — On titrating 1 gm. of sodium bicarbonate with normal hydrochloric acid, using methyl orange as the indicator, 11.9 cc. of the acid should be required.

1 cc. of normal HCl = 0.08405 gm. of $NaHCO_3$, log. 92454.

SODIUM BISULPHATE

(Sodium Acid Sulphate)

$NaHSO_4 + H_2O$. Mol. Wt. 138.13.

Colorless crystals, or white, fused pieces, easily soluble in water. The aqueous solution is strongly acid to litmus paper.

TESTS OF PURITY

Heavy Metals. — The solution of 1 gm. of sodium bisulphate in 20 cc. of water should not be affected by hydrogen sulphide water; nor should it show any change on making it alkaline with ammonia water, and then adding ammonium sulphide solution.

Chlorides. — 20 cc. of the 1:20 aqueous solution should not be rendered turbid on adding silver nitrate solution.

Arsenic. — The mixture of 1 gm. of powdered sodium bisulphate with 3 cc. of stannous chloride solution should not acquire a darker color within one hour.

Potassium. — The yellow-colored flame produced by sodium bisulphate, when observed through cobalt glass, should exhibit only a transient violet color.

Quantitative Determination. — Dissolve 1 gm. of sodium bisulphate in 50 cc. of water, and titrate with normal potassium hydroxide, using methyl orange as the indicator.

1 cc. of normal KOH = 0.13813 gm. of $NaHSO_4 + H_2O$, log. 14029.

SODIUM BISULPHITE

(Sodium Acid Sulphite)

$NaHSO_3$. Mol. Wt. 104.118.

A white powder, having an odor of sulphurous acid, and soluble in 4 parts of water. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Heavy Metals and Arsenic. — Completely evaporate a mixture of 5 gm. of sodium bisulphite with 5 cc. of pure sulphuric acid (sp. gr. 1.84) on the sand-bath, and dissolve the residue in 20 cc. of water. 10 cc. of this solution should not be affected by hydrogen sulphide water. On adding to the other 10 cc. of the solution a solution of ammonium molybdate in nitric acid, and heating the mixture to 70 to 80° C., the liquid should not acquire a yellow color, nor should a yellow precipitate form.

Quantitative Determination. — Dissolve 1 gm. of sodium bisulphite in thoroughly boiled water and dilute to 100 cc. Run this solution from a burette into a mixture of 30 cc. of decinormal iodine and 5 cc. of hydrochloric acid, with constant shaking, until complete decoloration ensues.

1 cc. of decinormal $\cdot$ I = 0.0052059 gm. of $NaHSO_3$, log. 71649.

SODIUM BORATE, CRYSTALS

(BORAX; SODIUM TETRABORATE; SODIUM BIBORATE)

$Na_2B_4O_7 + 10\ H_2O$. Mol. Wt. 382.26.

Hard, colorless crystals, or crystalline pieces, soluble in 17 parts of cold, and in 0.5 part of boiling, water, freely soluble in glycerin, but insoluble in alcohol. When borax is heated, it swells up, the water of crystallization being expelled, and at a red heat the anhydrous borax fuses to a transparent, colorless mass.

The aqueous solution is alkaline to litmus paper, and when acidulated with hydrochloric acid it colors turmeric paper brown. The brown color becomes particularly evident as the paper dries, and when moistened with ammonia water the color becomes greenish-black.

I

SODIUM BORATE

TESTS OF PURITY

Carbonates and Sulphates.—The solution of 1 gm. of borax in 20 cc. of water should not effervesce on the addition of hydrochloric acid, and should not be affected by the subsequent addition of barium chloride solution.

Chlorides.—On adding to 20 cc. of the 1:20 aqueous solution 3 cc. of nitric acid, followed by silver nitrate solution, at most a slight opalescent turbidity should develop.

Quantitative Determination.*—Dissolve 1 gm. of borax in 50 cc. of water, add 1 drop of methyl orange solution, and titrate with fifth normal hydrochloric acid.

1 cc. of fifth normal HCl = 0.038226 gm. of $Na_2B_4O_7 + 10H_2O$, log. 58235.

II

SODIUM BORATE, PUREST CRYSTALS

TESTS OF PURITY

Content of Water.†—1 gm. of borax, on ignition, should yield a residue weighing 0.529 gm.

Carbonates and Sulphates.—The solution of 1 gm. of borax in 20 cc. of water should not effervesce on adding 1 cc. of hydrochloric acid, nor should it become turbid on the subsequent addition of barium chloride solution.

Chlorides.—On adding to 20 cc. of the 1:20 aqueous solution 3 cc. of nitric acid, followed by silver nitrate solution, no change should appear.

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* Compare method of Schwartz as given in Sutton, Volumet. Anal., 9 ed., p. 94 (1904).

† Besides this prismatic borax containing 10 molecules of water of crystallization, there is another borax containing 5 molecules of water of crystallization, crystallizing in octahedra.

Calcium. — The solution of 1 gm. of borax in 20 cc. of water should not be rendered turbid by ammonium oxalate solution.

Iron and Other Metals. —

(*a*) The solution of 1 gm. of borax in 20 cc. of water should not be reddened on the addition of 2 cc. of hydrochloric acid, followed by potassium sulphocyanate solution.

(*b*) 20 cc. of the 1:20 aqueous solution, acidulated with 2 cc. of hydrochloric acid (sp. gr. 1.124), should remain unchanged on the addition of hydrogen sulphide water.

Quantitative Determination. — The determination is carried out as detailed under Sodium Borate.

SODIUM BORATE, PUREST, CALCINED.

A white, spongy mass, or white powder, containing at least 75 per cent of $Na_2B_4O_7$ (anhydrous).

TESTS OF PURITY

The tests to be made are those given under Sodium Borate, Purest, Cryst. But for each gram of crystallized borax use 0.7 gm. of the calcined.

Quantitative Determination.* — Dissolve 1 gm. of calcined borax in 50 cc. of water, add 1 drop of methyl orange solution, and titrate with fifth normal hydrochloric acid. At least 37.2 cc. of the acid should be necessary to produce the end-point.

1 cc. of fifth normal HCl = 0.02021 gm. of $Na_2B_4O_7$, log. 30557.

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* Compare methods given by Sutton, Volumet. Anal., 9 ed., p. 94 (1904).

SODIUM BORATE, PUREST, FUSED

(Borax Glass; Anhydrous Borax)

$Na_2B_4O_7$. Mol. Wt. 202.1.

Colorless, vitreous pieces, which take up moisture from the air, and in consequence become opaque.

TESTS OF PURITY

The tests to be made are those given under Sodium Borate, Purest, Cryst. But for each gram of the crystallized borax use 0.5 gm. of the fused.

Quantitative Determination. — This is carried out as described under Sodium Borate, Purest, Calcined.

SODIUM BROMATE

$NaBrO_3$. Mol. Wt. 151.01.

Shiny crystals, or crystalline powder, soluble in 3 parts of cold, and in about 1.5 parts of boiling, water.

TESTS OF PURITY

Bromides. — On dissolving 2 gm. of sodium bromate in 20 cc. of water, and adding 5 cc. of dilute sulphuric acid, the solution should not immediately acquire a yellow color.

Quantitative Determination. — Dissolve 0.10 to 0.15 gm. of sodium bromate, previously dried for twenty-four hours over sulphuric acid, in 20 cc. of water, add 3 gm. of potassium iodide and 5 cc. of hydrochloric acid, and titrate the liberated iodine with decinormal sodium thiosulphate.

1 cc. of decinormal $Na_2S_2O_3$ = 0.002517 gm. of $NaBrO_3$, log. 40088.

SODIUM CARBONATE

I

SODIUM CARBONATE, CRYSTALS

$Na_2CO_3 + 10H_2O$. Mol. Wt. 286.26.

Colorless, transparent crystals, efflorescent in the air, soluble in 1.6 parts of cold, and 0.2 part of boiling, water. The aqueous solution is strongly alkaline to litmus paper. Sodium carbonate is insoluble in alcohol. The crystallized sodium carbonate contains 37 per cent of the anhydrous salt, Na_2CO_3.

TESTS OF PURITY

Substances Insoluble in Water. — 20 gm. of sodium carbonate should completely dissolve in 80 cc. of water, yielding a perfectly colorless solution.

Sodium Hydroxide. — In a graduated flask of 100 cc. capacity, dissolve 3 gm. of crystallized sodium carbonate in 50 cc. of water, add to the solution 6 gm. of crystallized barium chloride dissolved in 30 cc. of water, and then fill with water up to the mark. After thoroughly shaking, filter, and to 50 cc. of the filtrate add some phenolphthalein solution. The liquid should not acquire a red color. Use boiled water throughout.

Sulphates. — On boiling a solution of 10 gm. of sodium carbonate in 50 cc. of water and 10 cc. of hydrochloric acid, for a few minutes, and then adding barium chloride solution, no precipitate of barium sulphate should form within twelve hours.

Chlorides. — The solution of 5 gm. of sodium carbonate in 50 cc. of water and 10 cc. of nitric acid should not be affected by silver nitrate solution.

Silicates. — Dissolve 20 gm. of crystallized sodium carbonate in 30 cc. of hydrochloric acid in a platinum dish, and evaporate the solution on the water-bath to dryness. Dry the residue half an hour at 120° C., and then dissolve it in 3 cc. of hydrochloric acid and 50 cc. of water. The solution should be perfectly clear.

Nitrates. — Dissolve 1 gm. of sodium carbonate in 10 cc. of dilute sulphuric acid, and overlay this liquid on 5 cc. of a solution of 0.5 gm. of diphenylamine in 100 cc. of concentrated sulphuric acid and 20 cc. of water. No blue color should form at the contact-surfaces of the two liquids.

Phosphates. — To the solution of 20 gm. of sodium carbonate in 50 cc. of nitric acid add 50 cc. of a solution of ammonium molybdate in nitric acid. No yellow precipitate should form in the liquid on standing two to three hours at about 40° C.

Potassium. — The yellow color imparted by sodium carbonate to the flame, when observed through cobalt glass, should at most have a transient violet tinge.

Ammonium Compounds. — On adding 1 cc. of Nessler's reagent to a solution of 10 gm. of sodium carbonate in 50 cc. of water, no change should be observed.

Calcium and Magnesium. — Dissolve 10 gm. of sodium carbonate in 10 cc. of water and 10 cc. of hydrochloric acid, and add 5 cc. of ammonia water, followed by ammonium oxalate solution. The liquid should remain perfectly clear, and should show no change even on the addition of ammonium phosphate solution and standing for several hours.

Heavy Metals. —

(*a*) The solution of 20 gm. of sodium carbonate in 50 cc. of water and 20 cc. of hydrochloric acid should not be affected by hydrogen sulphide water; on now adding to the solution 5 cc. of ammonia water and a few drops of ammonium sulphide solution, no turbidity or green color should develop.

(*b*) A solution of 10 gm. of sodium carbonate in 25 cc. of water and 10 cc. of hydrochloric acid should not acquire a red color on the addition of potassium sulphocyanate solution.

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen by adding dilute sulphuric acid (1:5). Dissolve 30 gm. of sodium carbonate in 100 cc. of dilute sulphuric acid (1:5), introduce the solution in small quantities at a time into the Marsh apparatus, and maintain a slow stream of gas for about half an hour. At the close of this period no deposit of arsenic should be visible within the reduction tube.

Quantitative Determination. — Dissolve 3 gm. of sodium carbonate in 50 cc. of water, and titrate with normal hydrochloric acid, using methyl orange as the indicator.

1 cc. of normal HCl = 0.14313 gm. of $Na_2CO_3 + 10H_2O$, log. 15573.

II

SODIUM CARBONATE, DRIED

A white, dry powder, containing about 80 per cent of Na_2CO_3.

TESTS OF PURITY

The tests to be made are those given under Sodium Carbonate Crystals. But for each gram of the crystals use 0.45 gm. of the dried.

Quantitative Determination. — Dissolve 1 gm. of the sodium carbonate in 50 cc. of water, and titrate with normal hydrochloric acid, using methyl orange as the indicator. At least 15 cc. of normal acid should be required.

1 cc. of normal HCl = 0.05305 gm. of Na_2CO_3, log. 72469.

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III

SODIUM CARBONATE, ANHYDROUS

Na_2CO_3. Mol. Wt. 106.1

A white powder, containing 99 to 100 per cent of Na_2CO_3.*

TESTS OF PURITY

The tests to be made are those given under Sodium Carbonate Crystals. But for each gram of crystals use 0.35 gm. of the anhydrous.

Quantitative Determination. — Dissolve 1 gm. in 50 cc. of water and titrate with normal hydrochloric acid, using methyl orange as indicator. At least 18.7 cc. of the normal acid should be required.

1 cc. of normal HCl = 0.05305 gm. of Na_2CO_3, log. 72469.

SODIUM CHLORIDE

NaCl. Mol. Wt. 58.5.

I

SODIUM CHLORIDE

White, cubical crystals, or crystalline powder, soluble in 2.7 parts of water. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — 3 gm. of sodium chloride yield a perfectly clear solution with 20 cc. of water. On diluting this solution with 80 cc. of water, adding 1 cc. of hydrochloric acid, heating to boiling, and then adding barium chloride solution, no precipitate of barium sulphate should form within twelve hours.

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* Sodium carbonate, which is to be used as a standard in volumetric determinations, must first be heated for thirty minutes in a sand-bath, or in an air-bath, at 270 to 300° C. (See Lunge, Ztschr. angew. Chem., **17**, 231 (1904); J. Chem. Soc., **86**, II, 289 (1904).)

Alkaline Earths, and Heavy Metals. — The solution of 3 gm. of sodium chloride in 50 cc. of water, heated to boiling, should not be affected by ammonium oxalate solution; nor by sodium carbonate solution; nor by ammonium sulphide solution.

Magnesium. — Dissolve 3 gm. of sodium chloride in 10 cc. of water, and add 5 cc. of ammonia water and ammonium phosphate solution. No precipitate should form within three hours.

Iodides. — On adding to 20 cc. of the 1:20 aqueous solution one drop of ferric chloride solution and some starch solution, no blue color should develop.

Potassium. — On adding platinic chloride solution and 3 cc. of alcohol (95 per cent) to the solution of 1 gm. of sodium chloride in 5 cc. of water, no precipitate should form within two hours.

Iron. — The solution of 3 gm. of sodium chloride in 20 cc. of water boiled with a few drops of nitric acid should not acquire a red color on the addition of potassium sulphocyanate solution.

Ammonium. — The solution of 3 gm. of sodium chloride in 20 cc. of water should not be changed in appearance on the addition of Nessler's reagent.

Quantitative Determination. — Dissolve 0.2 gm. of sodium chloride in 100 cc. of water, add a few drops of potassium chromate solution, and titrate with decinormal silver nitrate solution until a permanent red precipitate just begins to form.

1 cc. of decinormal $AgNO_3$ = 0.00585 gm. of NaCl, log. 76716.

II

SODIUM CHLORIDE, FUSED

Colorless, translucent pieces.

TESTS OF PURITY

The tests and the quantitative determination as given under Sodium Chloride are to be made.

SODIUM HYDROXIDE

(Caustic Soda; Sodium Hydrate)

NaOH. Mol. Wt. 40.058.

Three grades of sodium hydroxide are used in the chemical laboratory:

1. Sodium Hydroxide, from Sodium.
2. Sodium Hydroxide, Purified by Alcohol.
3. Sodium Hydroxide, Purified.

These three preparations vary chiefly in their content of chloride, sulphate, silica, and alumina.

I

SODIUM HYDROXIDE, FROM SODIUM

White pieces showing a crystalline structure on fracture. The preparation contains from 95 to 98 per cent of NaOH.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of sodium hydroxide in 50 cc. of water, acidulate with 15 cc. of hydrochloric acid, heat to boiling, and add barium chloride solution. No precipitate of barium sulphate should form within twelve hours.

Chlorides. — Dissolve 1 gm. of sodium hydroxide in 20 cc. of water, add 10 cc. of nitric acid and a few drops of silver nitrate solution. At most a slight opalescent turbidity should develop.

Nitrogen as Nitrates, Nitrites, Ammonia, etc. —

(*a*) To 2 gm. of sodium hydroxide dissolved in 10 cc. of water add 20 cc. of dilute sulphuric acid, 1 drop of

1:1000 indigo solution, a granule of sodium chloride, and then 10 cc. of concentrated sulphuric acid. The blue color of the mixture should not disappear within ten minutes.

(*b*) Dissolve 25 gm. of sodium hydroxide in 100 cc. of water in a distillation flask, and add 5 gm. of zinc dust and 5 gm. of iron by hydrogen. Connect the flask with a condenser and a U-tube receiver containing 3 to 5 cc. of fifth normal hydrochloric acid and 10 cc. of water, allow to stand for several hours, and then distil off about 25 cc. over a small flame. Titrate the distillate with fifth normal potassium hydroxide, using methyl orange as the indicator. At most 0.2 cc. of the acid should have been required to neutralize the ammonia.

Silica. — Dissolve 5 gm. of sodium hydroxide in 25 cc. of water and 25 cc. of hydrochloric acid in a platinum dish, and evaporate to dryness on a water-bath. Dry the residue for half an hour on the sand-bath at about 120° C., and then dissolve it in 10 cc. of hydrochloric acid and 90 cc. of water. Any insoluble residue should be filtered off, washed, and ignited. Its weight should not exceed 0.0005 gm.

Alumina, Calcium, and Heavy Metals. — 5 gm. of sodium hydroxide should afford a clear and colorless solution with 10 cc. of water. To this solution add 30 cc. of acetic acid (sp. gr. 1.041), followed by 10 cc. of ammonia water. Then dilute with 55 cc. of water, and heat the liquid on the water-bath until the odor of ammonia has disappeared. Add 2 to 3 drops of ammonia water, and allow to stand twelve hours. Within this time, no precipitate, or only a very slight flocculent precipitate, should form. In the latter case, the precipitate is filtered off and washed. Its weight upon ignition should not exceed 0.0005 gm. With the filtrate, the following tests are made:

To 50 cc. add a few cubic centimeters of ammonium oxalate solution. No precipitate of calcium oxalate should form on standing two hours.

To another 50 cc. add a few drops of ammonium sulphide solution; no change should appear.

Quantitative Determination and Determination of Sodium Carbonate Content. — Titrate the solution of 1 gm. of sodium hydroxide in 100 cc. of water with normal hydrochloric acid in the cold, using phenolphthalein as indicator. At least 24 cc. of normal acid should be required to destroy the red color. Now add 1 drop of methyl orange, and titrate further until the color changes to red. In the second titration, at most 0.3 cc. of acid should be used (3.18 per cent of Na_2CO_3).*

1 cc. of normal HCl = 0.04005 gm. of NaOH, log. 60260.

1 cc. of normal HCl = 0.05305 gm. of Na_2CO_3, log. 72469.

II

SODIUM HYDROXIDE, PURIFIED BY ALCOHOL

White, very hygroscopic sticks or pieces, exhibiting a crystalline fracture. The preparation contains 95 to 98 per cent of NaOH.

TESTS OF PURITY

Sulphates. — Dissolve 3 gm. of sodium hydroxide in 50 cc. of water, acidulate with 15 cc. of hydrochloric acid, heat to boiling, and add barium chloride solution. The liquid should not have more than a slight turbidity; and it should still be transparent when observed in a test-tube of 2 cm. diameter.

Chlorides. — The solution of 1 gm. of sodium hydroxide in 20 cc. of water and 10 cc. of nitric acid may be rendered opalescent on adding a few drops of silver nitrate solution, but should develop no precipitate.

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* Regarding the calculation of the sodium carbonate, see the note under Potassium Hydroxide, Purest.

Nitrogen as Nitrates, Nitrites, Ammonia, etc. — The test is carried out as detailed under Sodium Hydroxide, From Sodium.

Silica. — Dissolve 5 gm. of sodium hydroxide in 25 cc. of water with 25 cc. of hydrochloric acid in a platinum dish, and evaporate to dryness on the water-bath. Dry the residue on the sand-bath for half an hour at about 120° C., and then dissolve it in 10 cc. of hydrochloric acid and 90 cc. of water. Any insoluble residue is filtered off, washed, and ignited. Its weight after ignition should not exceed 0.0025 gm.

Alumina, Calcium, and Heavy Metals. — 5 gm. of sodium hydroxide should afford a clear and colorless solution with 20 cc. of water. Dilute the solution to 100 cc., and then add 30 cc. of acetic acid (sp. gr. 1.041) and 10 cc. of ammonia water. At most a slight turbidity should develop within five minutes, but no flocculent precipitate of aluminum hydroxide should form; nor should an immediate change in appearance occur on the addition of ammonium oxalate and ammonium sulphide solution.

Quantitative Determination. — This is carried out as described under Sodium Hydroxide, From Sodium. The amount of sodium carbonate present should not exceed 4 per cent.

III

SODIUM HYDROXIDE, PURIFIED

White, very hygroscopic sticks or pieces, exhibiting a crystalline fracture. The preparation contains 90 to 95 per cent of NaOH.

TESTS OF PURITY

Nitrogen as Nitrates and Nitrites. — To the solution of 2 gm. of sodium hydroxide in 10 cc. of water, add 15 cc. of 16 per cent sulphuric acid, 1 drop of 1:1000 indigo solution, a granule of sodium chloride, and then 10 cc. of concentrated sulphuric acid. The blue color of the mixture should not disappear within ten minutes.

Alumina, Calcium, and Heavy Metals. 2.5 gm. of sodium hydroxide should afford a clear and colorless solution with 10 cc. of water. Dilute the solution to 100 cc. and add 15 cc. of acetic acid (sp. gr. 1.041) and 5 cc. of ammonia water. At most a slight turbidity should develop within five minutes, but no flocks of aluminum hydroxide should separate. The solution thus tested should not immediately be rendered turbid on the addition of ammonium oxalate solution; and on adding to it ammonium sulphide solution, it should acquire at most a slight green color.

Quantitative Determination and Determination of the Sodium Carbonate Content.—Titrate a solution of 1 gm. of sodium hydroxide in 100 cc. of water with normal hydrochloric acid in the cold, using phenolphthalein as the indicator. At least 22.5 cc. of normal acid should be required to discharge the color. Now add 1 drop of methyl orange, and titrate further until the color again changes to red. In this second titration at most 0.5 cc. of the acid should be necessary (5.3 per cent of Na_2CO_3).*

1 cc. of normal HCl = 0.04005 gm. of NaOH, log. 60260.
1 cc. of normal HCl = 0.05305 gm. of Na_2CO_3, log. 72469

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* Regarding the calculation of the sodium carbonate, see the note under Potassium Hydroxide, Purest.

SODIUM HYROXIDE SOLUTIONS

(CAUSTIC SODA SOLUTIONS)

I

SODIUM HYDROXIDE, SOLUTION I—FREE FROM NITROGEN

A colorless liquid of specific gravity 1.3, and containing about 27 per cent of sodium hydroxide, NaOH.

TESTS OF PURITY

Nitrogen of Nitrates, Nitrites, Ammonia, etc.—The test for nitrogen is carried out by the distillation method as described under Sodium Hydroxide, From Sodium. For this purpose 70 cc. (90 gm.) of sodium hydroxide, solution I, should be taken.

II

SODIUM HYDROXIDE, SOLUTION II—FREE FROM NITROGEN

A colorless liquid of specific gravity 1.3 and containing about 27 per cent of sodium hydroxide, NaOH.

TESTS OF PURITY

The tests to be made are those given under Sodium Hydroxide, Purified by Alcohol. But for each gram of sodium hydroxide, purified by alcohol, use 2.9 cc. (3.7 gm.) of sodium hydroxide, solution II, N-free.

III

SODIUM HYDROXIDE, SOLUTION III. SP. GR. 1.168

A clear, colorless liquid, of specific gravity 1.168 to 1.172, and containing about 15 per cent of sodium hydroxide, NaOH.

TESTS OF PURITY

The tests to be made are those given under Sodium Hydroxide, Purified by Alcohol. But for each gram of sodium hydroxide, purified by alcohol, use 5.6 cc. (6.5 gm.) of sodium hydroxide, solution III.

SODIUM HYDROXIDE WITH LIME

(SODA-LIME)

I

SODIUM HYDROXIDE WITH LIME

A white, porous mass, finely or coarsely granulated.

TESTS OF PURITY

Excess of Carbonate. — Soda-lime, when treated with dilute sulphuric acid, should not effervesce strongly.

Nitrogen. —

(*a*) On igniting soda-lime in a test-tube, it should develop no vapors of ammonia (to be ascertained by moistened litmus paper).

(*b*) Shake 50 gm. of finely powdered soda-lime with a mixture of 5 gm. of zinc dust and 5 gm. of iron by hydrogen in 200 cc. of water in a flask of about 500 cc. capacity. Connect the flask with a condenser and a receiver containing 2 to 3 cc. of fifth normal hydrochloric acid, allow to stand for two to three hours, and then distil off about 25 cc. over a small flame. Now titrate the distillate with fifth normal potassium hydroxide, using methyl orange as the indicator. No more than 0.2 cc. of the acid should have been required to neutralize the ammonia.

II

SODIUM HYDROXIDE WITH LIME FROM ICELAND SPAR

A white, porous, granular mass.

TESTS OF PURITY

Chlorides. — Dissolve 5 gm. of soda-lime in 50 cc. of nitric acid, and dilute the solution with 100 cc. of water. The liquid should acquire at most a slight opalescent turbidity on the addition of silver nitrate solution.

Phosphorus. — Dissolve 10 gm. of soda-lime in 100 cc. of nitric acid, and add to the solution 25 cc. of a solution of ammonium molybdate in nitric acid. No yellow precipitate should form within two to three hours on standing at about 40° C.

Sulphur. — Thoroughly mix 5 gm. of finely powdered soda-lime with 2 gm. of sodium nitrate, and ignite the mixture in a silver crucible. Dissolve the melt in 50 cc. of water with 20 cc. of hydrochloric acid, filter, and to the filtrate add barium chloride solution. No precipitate of barium sulphate should form within twelve hours.

SODIUM NITRATE

$NaNO_3$. Mol. Wt. 85.09.

Colorless, transparent, rhombohedric crystals, soluble in 1.2 parts of water and in 50 parts of alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Sulphates. — On dissolving 3 gm. of sodium nitrate in 60 cc. of water, and adding 0.5 cc. of hydrochloric acid followed by barium chloride solution, no precipitate of barium sulphate should form on standing twelve hours.

Chlorides. — The solution of 1 gm. of sodium nitrate in 20 cc. of water, acidulated with a few drops of nitric acid, should not be rendered turbid on the addition of silver nitrate solution.

Chlorates and Perchlorates. — On gently igniting 1 gm. of sodium nitrate, dissolving the residue in 20 cc. of water, and adding 1 cc. of nitric acid, the solution should not be rendered turbid on adding silver nitrate solution.

Calcium and Heavy Metals. —

(*a*) The solution of 3 gm. of sodium nitrate in 50 cc. of water should show no change on the addition of hydrogen sulphide water.

(*b*) The solution of 3 gm. of sodium nitrate in 50 cc. of water should not be changed in appearance by the addition of ammonia water nor by adding ammonium oxalate and ammonium sulphide solutions.

Iron. — 20 cc. of the 1:20 aqueous solution, acidulated with 1 cc. of hydrochloric acid, should not be reddened on the addition of potassium sulphocyanate solution.

Nitrites. — Dissolve 1 gm. of sodium nitrate in 20 cc. of water, and add 1 cc. of dilute sulphuric acid and 1 cc. of a 1:200 solution of metaphenylenediamine hydrochloride.* No yellow or yellowish-brown color should develop.

Iodates and Nitrites. — To 5 cc. of the 1:20 aqueous solution add 3 or 4 drops of dilute sulphuric acid and zinc iodide-starch solution. No immediate blue color should develop.

Potassium. —

(*a*) The color imparted to the flame by sodium nitrate, when observed through cobalt glass, should be pure yellow, without violet; or at most only momentarily violet.

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* Should the solution of metaphenylenediamine hydrochloride have a color, it should be decolorized before use by warming with ignited animal charcoal.

(*b*) Dissolve 3 gm. of sodium nitrate with the aid of heat in 6 cc. of water, and to 3 cc. of the filtered solution add 1 or 2 drops of a solution of cobalt and sodium nitrite in acetic acid.* The liquid should not become turbid, and on standing two hours at about 50° C., no yellow precipitate should form.

SODIUM NITRITE

$NaNO_2$. Mol. Wt. 69.09.

I

SODIUM NITRITE

White, or very slightly yellowish, tough sticks, easily and clearly soluble in water. The aqueous solution is slightly alkaline to litmus paper. The preparation contains 98 to 99 per cent of $NaNO_2$.

TESTS OF PURITY

Chlorides. — The solution of 1 gm. of sodium nitrite in 20 cc. of water should not acquire more than a slight opalescent turbidity on the addition of 5 cc. of nitric acid and some silver nitrate solution.

Sulphates. — 20 cc. of the 1 : 20 aqueous solution, acidulated with 5 cc. of nitric acid, should not be rendered turbid on the addition of barium nitrate solution.

Potassium. — The color imparted to the flame by sodium nitrite, when observed through cobalt glass, should be pure yellow, without violet; or at most only momentarily violet.

Heavy Metals. — 20 cc. of the 1 : 20 aqueous solution should show no change on the addition of a few drops of ammonium sulphide solution.

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* The acetic acid solution of cobalt and sodium nitrite for testing for potassium salts is prepared as follows:

To a solution of 10 gm. of crystallized cobalt acetate in 25 cc. of water, add a solution of 20 gm. of pure sodium nitrite in 40 to 50 cc. of water previously acidulated with a little acetic acid. Allow the solution to stand two hours at about 50° C., and then filter.

Quantitative Determination. Dissolve 1 gm. of sodium nitrite in water and dilute to 100 cc. Make a mixture of 50 cc. of decinormal potassium permanganate (accurately measured) with 300 cc. of water and 25 cc. of dilute sulphuric acid, warm to 40 or 50° C., and, while constantly shaking, allow the nitrite solution to run slowly into it until the red color disappears. Care must be taken towards the end to add the nitrite solution very slowly, because the change from red to colorless always requires some time. Not more than 17.6 cc. of the sodium nitrite solution should be required.

1 cc. of decinormal $KMnO_4$ = 0.0034545 gm. of $NaNO_2$, log. 53838.

II

SODIUM NITRITE, FREE FROM POTASSIUM

TESTS OF PURITY

Potassium. — To a solution of 5 gm. of crystallized cobalt acetate in 12 cc. of water add a solution of 10 gm. of sodium nitrite in 20 cc. of water previously acidulated with 2 cc. of diluted acetic acid. Dilute the solution with 25 cc. of water and allow it to stand twenty-four hours at 40 to 50° C. No yellow precipitate should form within this time.

Other Tests. — The other tests and the quantitative determination given under Sodium Nitrite should also be made observing the conditions there described.

SODIUM NITROFERRICYANIDE

(SODIUM NITROPRUSSIDE)

$Na_2Fe(CN)_5(NO) + 2H_2O$. Mol. Wt. 298.27.

Ruby-red, transparent crystals, affording a clear solution with 2.5 parts of water; also soluble in alcohol.

TEST OF PURITY

Sulphate. The solution of 1 gm. of sodium nitroprusside in 50 cc. of water acidulated with 1 cc. of hydrochloric acid should not be rendered turbid on adding barium chloride solution.

SODIUM OXALATE

(SÖRENSEN'S OXALATE)

$Na_2C_2O_4$. Mol. Wt. 134.1.

A white, crystalline powder, soluble in 31 parts of cold, and in 16 parts of boiling, water. Sodium oxalate which is to be used in acidimetric and oxidimetric determinations as a standard, is obtained by precipitation with alcohol and drying at 240° C. The preparation contains 100 per cent of $Na_2C_2O_4$.

TESTS OF PURITY

Hygroscopic Moisture.* — 10 gm. of sodium oxalate, when dried in a water-drying oven for twenty-four hours, should not lose more than 0.001 gm. in weight.

Sodium Carbonate, Sodium Binoxalate. — Introduce into a conical Jena flask about 250 cc. of water and 10 drops of phenolphthalein solution (0.5 gm. phenolphthalein dissolved in 50 cc. of alcohol and 50 cc. of water), and evaporate to about 180 cc. while passing in a current of pure air, free from carbon dioxide. Allow to cool to the ordinary temperature, add 5 gm. of sodium oxalate, shake carefully, and maintain the current of air. The oxalate slowly dissolves. If the solution is red, not more than 4 drops of decinormal acid should be required to render it colorless. But, if the solution is colorless, it must acquire a distinct red color on the addition of at most 2 drops of decinormal sodium hydroxide.

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* These tests are taken from the original paper by S. P. L. Sörensen, Ztschr. anal. Chem., **42**, 512 (1903); J. Chem. Soc., **84**, II, 684, 750 (1903).

Chlorides and Sulphates. — Decompose 10 gm. of sodium oxalate by heating in a platinum crucible, best over an alcohol lamp (illuminating gas contains sulphur). The carbonate formed is dissolved in nitric acid, and the solution filtered off from the carbon. On adding silver nitrate solution to half of the filtrate, no reaction for hydrochloric acid should be obtained; and in the other half no reaction for sulphuric acid should be obtained on adding barium nitrate solution.

Iron and Potassium. — Decompose 10 gm. of sodium oxalate by igniting moderately in a platinum crucible, removing the last traces of carbon by cautious ignition with a blast lamp. The residue, when treated with warm water in a platinum dish, should be completely soluble, and should leave at most a scarcely weighable trace of undissolved iron oxide. The solution is filtered if necessary, and supersaturated with hydrochloric acid as free from iron as possible. It is then evaporated in a platinum dish on the water-bath, and the residue dried for two hours in the drying oven at 120° C. The residue must dissolve clear in water and the solution should give:

(*a*) With potassium sulphocyanate solution only a very faint reaction for iron after boiling with a little nitric acid; and

(*b*) With sodium and cobalt nitrite solution, no reaction for potassium.

Foreign Organic Bodies. — In a clean, thoroughly ignited test-tube, heat 1 gm. of sodium oxalate with 10 cc. of pure concentrated sulphuric acid, as long as evolution of gas occurs, at first gently, and then more strongly, until vapors of sulphuric acid begin to be evolved. When cooled, the color of the sulphuric acid is compared with that of another portion of 10 cc. of sulphuric acid similarly treated, but without the addition of sodium oxalate. The sodium oxalate, treated in the manner described, should impart to the sulphuric acid not more than an exceedingly faint brownish tinge.

Quantitative Determination. — 0.4 to 0.5 gm. of the sodium oxalate dried to constant weight at 100° C. is cautiously heated in a covered platinum crucible. In order to avoid the error due to the sulphur in illuminating gas, the crucible is held in a sheet of asbestos provided with a round hole, or a Berzelius alcohol lamp is used. The conversion of the oxalate into carbonate is completed in from fifteen to thirty minutes; any carbon present is then burned off by more strongly heating the crucible while half covered. When cold, dissolve the contents of the crucible in water, and titrate in the cold with fifth normal hydrochloric acid, using methyl orange as the indicator.

1 cc. of fifth normal HCl = 0.01341 gm. of $Na_2C_2O_4$, log. 12743.

Literature: S. P. L. Sörensen, Ztschr. anal. Chem., **36**, 639 (1897); J. Chem. Soc., **74**, II, 185 (1898). Ztschr. anal. Chem., **42**, 333 (1903); J. Chem. Soc., **84**, II, 684 (1903). Ztschr. anal. Chem., **42**, 512 (1903); J. Chem. Soc., **84**, II, 684, 750 (1903). Ztschr. anal. Chem., **44**, 156 (1905); J. Chem. Soc., **88**, II, 415 (1905).

G. Lunge, Ztschr. angew. Chem., **17**, 230, 269 (1904); J. Chem. Soc., **86**, II, 289 (1904).

SODIUM PEROXIDE

(Sodium Superoxide)

Na_2O_2. Mol. Wt. 78.10.

A light, yellow powder, very easily soluble in water with the copious evolution of oxygen and great elevation of temperature. On cautiously adding sodium peroxide to a cooled, dilute mineral acid, hydrogen peroxide results. The preparation contains at least 95 per cent of Na_2O_2.

TESTS OF PURITY

Sulphates. — Add 5 gm. of sodium peroxide in small quantities at a time to a mixture of 25 cc. of hydrochloric acid and 100 cc. of water. The clear solution should develop no precipitate with barium chloride solution on standing twelve hours.

Halogens. — Add 3 gm. of sodium peroxide in small quantities at a time to a mixture of 20 cc. of nitric acid and 100 cc. of water. The clear liquid should exhibit at most a slight opalescent turbidity on the addition of silver nitrate solution.

Phosphates. — Add 2.5 gm. of sodium peroxide in small quantities at a time to a mixture of 20 cc. of nitric acid and 100 cc. of water. On now adding 25 cc. of a solution of ammonium molybdate in nitric acid and heating to 30 to 40° C., no yellow precipitate should form within two hours.

Nitrogen. — Mix 1 gm. of sodium peroxide with 0.2 gm. of grape sugar, very cautiously, in a capacious nickel crucible. Effect the deflagration of the mixture by very gently heating the bottom of the covered crucible. When cold, dissolve the residue in 5 cc. of water, acidulate with 10 cc. of dilute sulphuric acid, and overlay a few cubic centimeters of this solution on 5 cc. of diphenylamine solution (see Diphenylamine). No blue color should develop at the contact surfaces of the two liquids.

Heavy Metals. — On cautiously adding 5 gm. of sodium peroxide to 100 cc. of water, a perfectly clear and almost colorless solution should result.

40 cc. of this solution, acidulated with 10 cc. of hydrochloric acid, should not be affected by hydrogen sulphide water.

40 cc. of the alkaline solution should develop no precipitate on the addition of a few drops of ammonium sulphide solution, nor should the liquid acquire a brown or a green color.

Quantitative Determination. — The quantitative determination of sodium peroxide is most simply effected by decomposing it in a Lunge gas volumeter,[*] using cobalt nitrate as a catalyzer and measuring the volume of oxygen evolved.

Introduce 0.5 gm. of sodium peroxide in a weighing flask into the outer compartment of the decomposition flask, and in the inner place a mixture of 15 cc. of dilute sulphuric acid and 2 or 3 drops of a saturated solution of cobalt nitrate. On tilting the flask, the liquid is allowed to mix with the sodium peroxide, whereby the decomposition of the latter is effected.

1 cc. of O at 0° C. and 760 mm. = 0.006964 gm. of Na_2O_2, log. 84286.

SODIUM PHOSPHATE

(DISODIUM HYDROGEN PHOSPHATE; SECONDARY SODIUM PHOSPHATE)

$Na_2HPO_4 + 12 H_2O$. Mol. Wt. 358.3.

Colorless, transparent crystals, efflorescent in dry air, and soluble in 6 parts of water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Carbonates and Sulphates.—20 cc. of the 1:20 aqueous solution should not effervesce on the addition of 1 cc. of hydrochloric acid; nor should it exhibit any change on the subsequent addition of barium chloride solution, on standing three hours.

Chlorides.—The solution of 1 gm. of sodium phosphate in 20 cc. of water, acidulated with 2 cc. of nitric acid, should show at most a very slight opalescent turbidity on the addition of silver nitrate solution.

Nitrates.—To a solution of 2 gm. of sodium phosphate in 10 cc. of water add 5 cc. of dilute sulphuric acid, 1 drop of 1:1000 indigo solution, a granule of sodium chloride, and

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[*] Lunge, Chem. Tech. Untersuch. Meth., 4 ed., **1**, 158 (1904).

10 cc. of concentrated sulphuric acid. The blue color of the liquid should not disappear within ten minutes.

Heavy Metals. — The solution of 2 gm. of sodium phosphate in 20 cc. of water, acidulated with 1 cc. of hydrochloric acid, should appear unchanged on the addition of hydrogen sulphide water. On now adding to the liquid 5 cc. of ammonia water and a few drops of ammonium sulphide solution, no precipitate should form, nor should a green color develop.

Arsenic. — Into the generating flask of a Marsh apparatus introduce 10 gm. of arsenic-free, metallic zinc, and start the hydrogen with sulphuric acid (1:5). Dissolve 2 gm. of sodium phosphate in 50 cc. of water, introduce the solution in small portions at a time into the Marsh apparatus, and maintain a slow stream of the gas for about one hour. No deposit of arsenic should be visible in the reduction tube within this time.

Potassium. — The color imparted by sodium phosphate to a colorless flame, when observed through cobalt glass, should not be tinged with violet, or should be only momentarily so.

SODIUM PYROPHOSPHATE

$Na_4P_2O_7 + 10\ H_2O$. Mol. Wt. 446.36.

Colorless, transparent crystals, soluble in 10 to 12 parts of cold water, and in somewhat more than 1 part of boiling water, but insoluble in alcohol. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Phosphates. — The 1:20 aqueous solution of sodium pyrophosphate should give a pure white precipitate with silver nitrate solution.

Other tests. — The tests as given under Sodium Phosphate are also to be made.

SODIUM SULPHATE

$Na_2SO_4 + 10H_2O$. Mol. Wt. 322.32.

Colorless, efflorescent crystals, soluble in 3 parts of cold, and in 0.4 part of boiling, water, but insoluble in alcohol. The aqueous solution is neutral to litmus paper.

TESTS OF PURITY

Substances Insoluble in Water. — 5 gm. of sodium sulphate should afford a clear solution with 50 cc. of water.

Chlorides, Heavy Metals, Calcium, and Magnesium. — 20 cc. portions of the 1:20 aqueous solution should not be affected by hydrogen sulphide water, ammonium oxalate, silver nitrate, and sodium phosphate solutions.

Iron. — 1 gm. of sodium sulphate dissolved in 20 cc. of water and boiled with a few drops of nitric acid should remain colorless on the addition of potassium sulphocyanate solution.

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen with sulphuric acid (1:5). Dissolve 2 gm. of sodium sulphate in 20 cc. of water, introduce the solution in small portions at a time into the Marsh apparatus, and maintain a slow stream of gas for about half an hour. No deposit of arsenic should be visible in the reduction tube within this time.

SODIUM SULPHIDE

$Na_2S + 9H_2O$. Mol. Wt. 240.20.

Colorless, transparent crystals, easily and clearly soluble in water. The aqueous solution is alkaline to litmus paper. The preparation contains at least 97 per cent of $Na_2S + 9H_2O$.

TESTS OF PURITY

Ammonium Salts. — On dissolving 3 gm. of sodium sulphide in 20 cc. of water, and heating with sodium hydroxide solution, no vapors of ammonia should be evolved (to be ascertained by moistened litmus paper).

Quantitative Determination; Sulphites, and Thiosulphates. —

(*a*) Dissolve 1 gm. of sodium sulphide in water and dilute to 100 cc. Allow 20 cc. of this solution to run into a mixture of 20 cc. of decinormal iodine and 3 cc. of hydrochloric acid which has been diluted with 100 cc. of water. Titrate the excess of iodine with decinormal sodium thiosulphate, using starch solution as the indicator.

1 cc. of decinormal I = 0.012015 gm. of $Na_2S + 9H_2O$, log. 07972.

(*b*) To 100 cc. of a solution of 1 gm. of sodium sulphide in sufficient water to make 100 cc., add 2 gm. of crystallized zinc sulphate dissolved in 150 cc. of water; shake vigorously, allow to stand half an hour, and then filter. Titrate 50 cc. of the filtrate with decinormal iodine, using starch solution as the indicator. Not more than 0.1 cc. of decinormal iodine should be required.

SODIUM SULPHIDE SOLUTION

Sodium sulphide solution contains 5 per cent of Na_2S and is used for determination of nitrogen according to Kjeldahl.

TEST OF PURITY

Nitrogen. — The test for nitrogen is to be carried out as described under Potassium Sulphide Solution.

SODIUM SULPHITE

I

SODIUM SULPHITE, CRYSTALS

$Na_2SO_3 + 7H_2O$. Mol. Wt. 252.27.

Colorless, prismatic crystals, efflorescent in air, and easily soluble in water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Heavy Metals and Arsenic. — The tests are carried out as detailed under Sodium Bisulphite.

Quantitative Determination. — Dissolve 1 gm. of sodium sulphite in boiled water and dilute to 100 cc. Allow the solution to run from a burette into a mixture of 30 cc. of decinormal iodine and 5 cc. of hydrochloric acid, with constant agitation, until complete decolorization occurs.

1 cc. of decinormal I = 0.0126136 gm. of $Na_2SO_3 + 7H_2O$, log. 10084.

II

SODIUM SULPHITE, DRIED

A white powder, containing 85 to 90 per cent of Na_2SO_3.

The tests and quantitative determination as given under Sodium Sulphite Crystals are to be made.

1 cc. of decinormal I = 0.006308 gm. of Na_2SO_3, log. 79989.

SODIUM THIOSULPHATE

(SODIUM HYPOSULPHITE)

$Na_2S_2O_3 + 5H_2O$. Mol. Wt. 248.3.

Colorless and odorless crystals, permanent in the air at ordinary temperatures. Sodium thiosulphate liquefies at 50° C. in its own water of crystallization, and is soluble in less than 1 part of cold water. The 1:1 aqueous solution is slightly alkaline to litmus paper.

TESTS OF PURITY

Carbonates, Sulphates, and Sulphites. — To a solution of 3 gm. of sodium thiosulphate in 50 cc. of water, add decinormal iodine solution (about 120 cc.) until the liquid has a slight yellow color; on now adding barium chloride solution, no turbidity should ensue.

Free Alkali. — The solution of 1 gm. of sodium thiosulphate in 10 cc. of water should not be reddened by phenolphthalein.

Sulphides. — The solution of 1 gm. of sodium thiosulphate in 10 cc. of water should not change in appearance on the addition of zinc sulphate solution.

Calcium. — The solution of 1 gm. of sodium thiosulphate in 20 cc. of water should not be rendered turbid on the addition of ammonia water and ammonium oxalate solution.

Quantitative Determination. — Dissolve 1 gm. of sodium thiosulphate in 100 cc. of water, and titrate with decinormal iodine solution, using starch solution as the indicator.

1 cc. of decinormal I = 0.02483 gm. of $Na_2S_2O_3 + 5H_2O$, log. 39498.

SODIUM TUNGSTATE

(Sodium Wolframate)

$Na_2WO_4 + 2H_2O$. Mol. Wt. 330.13.

Colorless prisms or rhombic plates, soluble in 4 parts of water. The aqueous solution is alkaline to litmus paper.

TESTS OF PURITY

Water. — After gently igniting 1 gm. of sodium tungstate, the residue should weigh 0.88 gm.

Chlorides. — Dissolve 1 gm. of sodium tungstate in 20 cc. of water, add 5 cc. of nitric acid, and filter. The filtrate should acquire at most a slight opalescent turbidity within ten minutes after adding a few drops of silver nitrate solution.

Sulphates. Dissolve 1 gm. of sodium tungstate in 25 cc. of water, add 5 cc. of nitric acid, boil for ten to fifteen minutes, and then filter. 20 cc. of the filtrate should not immediately be rendered turbid on the addition of barium nitrate solution.

Quantitative Determination.—Dissolve 1 gm. of sodium tungstate in 10 cc. of water, add 10 cc. of hydrochloric acid, evaporate on the water-bath to dryness, and heat the residue for half an hour at 120° C. Treat the residue with 20 cc. of hydrochloric acid, and repeat the entire operation three or four times. Finally digest the residue with ammonium nitrate solution to which a little nitric acid has been added, filter, wash the tungstic acid with dilute nitric acid, dry, ignite, and weigh the residue. The latter should weigh 0.69 gm.

SODIUM AND AMMONIUM PHOSPHATE

(Microcosmic Salt)

$NH_4NaHPO_4 + 4H_2O$. Mol. Wt. 209.19.

Colorless, monoclinic crystals, soluble in 5 parts of water. The aqueous solution is alkaline to litmus paper. Sodium and ammonium phosphate when fused on a platinum wire should yield a clear and colorless bead.

TESTS OF PURITY

The tests given under Sodium Phosphate are to be made, observing the conditions there described.

SODIUM AND POTASSIUM CARBONATE, FUSED, ANHYDROUS

White sticks used for making carbon dioxide free from air, according to Kreussler.

THYMOL

$C_6H_3(CH_3)(OH)(C_3H_7)1:3:4$. Mol. Wt. 150.11.

Colorless, hexagonal crystals with an ethereal odor, easily soluble in alcohol, ether, and chloroform, but difficultly soluble in water (1:1100). Thymol melts at 50 to 51° C., and boils at 228 to 230° C.

TESTS OF PURITY

Inorganic Matter. — 1 gm. of thymol on ignition should leave no weighable residue.

Free Acids. — Blue litmus paper should not be reddened by the aqueous or alcoholic solution.

Phenol. — The 1:1100 aqueous solution may exhibit a milky turbidity on the addition of bromine water, but should show no crystalline precipitate. Nor should the solution afford a violet color with ferric chloride.

TIN

Sn. Atomic Wt. 119.

A soft, almost silver-white metal, melting at 231° C. Tin dissolves in hot hydrochloric acid with the formation of stannous chloride. Hot concentrated nitric acid converts it into insoluble meta-stannic acid.

TESTS OF PURITY

Lead, Copper, Iron, and Zinc. — Digest 5 gm. of the tin with 40 cc. of nitric acid on the water-bath until the conversion of the metal into a white powder is complete. Then evaporate completely, stir the residue with 10 cc. of nitric acid and 50 cc. of water, and filter. To the filtrate add 1 cc. of dilute sulphuric acid, and evaporate on the water-bath as far as possible, and then take up the residue with 10 cc. of water. No weighable insoluble residue should remain (Pb).

If necessary, filter, and to the filtrate add ammonia water to alkaline reaction. The liquid should not acquire a blue color (Cu). Now add ammonium sulphide solution, and allow the mixture to stand for four to five hours at about 50° C. Should any precipitate form, its weight after ignition should not exceed 0.002 gm. (Fe, Zn).

TIN CHLORIDE

(STANNOUS CHLORIDE)

$SnCl_2 + 2H_2O$. Mol. Wt. 225.93.

Colorless crystals, easily and completely soluble in alcohol, and in water acidulated with hydrochloric acid. Stannous chloride solution is decomposed by much water, with the separation of a basic stannous chloride.

TESTS OF PURITY

Sulphates. Dissolve 1 gm. of stannous chloride in 5 cc. of hydrochloric acid (sp. gr. 1.19), and dilute with 50 cc. of water. The solution should not be affected by barium chloride solution.

Ammonium Compounds. On heating 1 gm. of stannous chloride with 10 cc. of sodium hydroxide solution (sp. gr. 1.3), no vapors of ammonia should be evolved (to be ascertained by moist litmus paper).

Earths, Alkalies, and Iron. Dissolve 2 gm. of stannous chloride in 10 cc. of hydrochloric acid (sp. gr. 1.19), dilute the solution with 100 cc. of water, and pass into the solution hydrogen sulphide gas until all the tin has been precipitated. Filter off the precipitate, evaporate the filtrate, and ignite the residue; the weight of the ignited residue should not exceed 0.001 gm. On heating the ignited residue with 1 cc. of hydrochloric acid, then diluting with 20 cc. of water, and adding potassium sulphocyanate solution, at most only a slight red color should develop.

Arsenic. — Boil 2 gm. of stannous chloride with 10 cc. of hydrochloric acid (sp. gr. 1.19) for several minutes. The liquid should remain clear and colorless for one hour.

Quantitative Determination. — Dissolve 0.5 gm. of stannous chloride in 2 cc. of hydrochloric acid (sp. gr. 1.19) and dilute the solution with 50 cc. of water. Then add 5 gm. of potassium and sodium tartrate, and sodium bicarbonate until the solution is alkaline to litmus paper. Now titrate with decinormal iodine, using starch solution as the indicator.

1 cc. of decinormal I = 0.0112966 gm. of $SnCl_2 + 2H_2O$, log. 05294.

TIN CHLORIDE SOLUTION

(Stannous Chloride Solution)

A slightly yellow, very refractive liquid, the specific gravity of which should not be less than 1.90. It is obtained by stirring 5 parts of stannous chloride with 1 part of hydrochloric acid, and then saturating the mixture with dry hydrochloric acid gas.

TESTS OF PURITY

Substances Precipitated by Alcohol. — On mixing stannous chloride solution with 10 times its volume of 85 per cent alcohol, no turbidity should ensue on standing for one hour.

Sulphuric Acid. — 5 cc. of stannous chloride solution diluted with 50 cc. of water should not be rendered turbid on the addition of barium chloride solution.

URANIUM ACETATE, FREE FROM SODIUM

(URANYL ACETATE)

$UO_2(C_2H_3O_2)_2 + 2H_2O$. Mol. Wt. 424.58.

A yellow, crystalline powder, easily soluble in water. As the preparation nearly always contains some basic salt, a clear solution is obtained only on adding a little acetic acid.

TESTS OF PURITY

Sulphates. The solution of 1 gm. of uranium acetate in 20 cc. of water and 2 to 3 cc. of dilute acetic acid should not be affected by barium chloride solution.

Sodium. Dissolve 5 gm. of uranium acetate in 200 cc. of water, with the aid of 10 cc. of dilute acetic acid. Add to the boiling solution an excess of ammonia water, filter, and evaporate the filtrate to dryness. Ignite the residue, dissolve it in water, and then titrate with normal hydrochloric acid, using methyl orange as the indicator. Not more than 0.1 cc. of normal hydrochloric acid should be required to produce the red color.

Earths. The solution of 1 gm. of uranium acetate in 20 cc. of water and 2 to 3 cc. of dilute acetic acid should remain clear on adding ammonia water and ammonium carbonate solution in excess.

Uranous Salt. The solution of 1 gm. of uranium acetate in 20 cc. of water and 1 cc. of dilute sulphuric acid should be colored red on the addition of 0.1 to 0.2 cc. of decinormal potassium permanganate.

Foreign Metals.

(*a*) On heating to boiling a solution of 5 gm. of uranium acetate in 100 cc. of water and 5 cc. of hydrochloric acid, no change should appear on passing into the solution hydrogen sulphide gas.

(*b*) On adding to the liquid obtained under the test for earths, 2 or 3 drops of ammonium sulphide solution, no dark-brown color should develop, nor should a precipitate form.

URANIUM NITRATE

(Uranyl Nitrate)

$UO_2(NO_3)_2 + 6H_2O$. Mol. Wt. 502.67.

Yellow crystals, having a greenish luster by reflected light, and efflorescing superficially in dry air. Uranium nitrate is easily soluble in water, alcohol, and ether. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Sulphates. — The 1:20 aqueous solution should not develop a turbidity within fifteen minutes on adding barium chloride solution.

Alkali Salts. — On igniting 1 gm. of uranium nitrate, treating the residue with 20 cc. of water, filtering, and evaporating the filtrate, no weighable residue should remain.

Earths. — The solution of 1 gm. of uranium nitrate in 20 cc. of water should remain clear on the addition of ammonia water and ammonium carbonate solution in excess.

Uranous Salt. — The solution of 1 gm. of uranium nitrate in 20 cc. of water and 1 cc. of dilute sulphuric acid should be colored red on the addition of 0.1 to 0.2 cc. of decinormal potassium permanganate.

Foreign Metals. —

(*a*) The liquid obtained under the test for earths, when treated with 2 or 3 drops of ammonium sulphide solution, should not acquire a dark-brown color, nor should it develop a precipitate.

(*b*) On heating to boiling a solution of 5 gm. of uranium nitrate in 100 cc. of water and 5 cc. of hydrochloric acid, no change should appear on passing hydrogen sulphide gas into the solution.

WATER DISTILLED

H_2O. Mol. Wt. 18.01.

Distilled water must be neutral to litmus paper.

TESTS OF PURITY

Ammonia and Ammonium Compounds. — 50 cc. of the water should show no change on the addition of 10 to 15 drops of Nessler's reagent.

Chlorides. 100 cc. of the water should show no change on adding a few drops of nitric acid followed by silver nitrate solution.

Sulphates. On adding 0.25 cc. of hydrochloric acid and some barium chloride solution to 100 cc. of water, no precipitate of barium sulphate should form on standing twelve hours.

Nitrates. Introduce 5 cc. of diphenylamine solution (see Diphenylamine) into a test tube, and overlay it with 10 cc. of water. No blue color should form at the contact-surfaces of the two liquids.

Non-volatile Matter. — 100 cc. of the water evaporated on the water bath should leave no weighable residue.

Heavy Metals and Calcium. — 100 cc. of the water should show no change with hydrogen sulphide water, ammonia water, ammonium sulphide, and ammonium oxalate solutions.

Substances Oxidizable by Permanganate (Organic Matter, Nitrites, etc.). Heat to boiling 100 cc. of the water with 1 cc. of 16 per cent sulphuric acid, add 1 drop of potassium permanganate solution (1:1000), and maintain the boiling for three minutes. The liquid should not be decolorized.

XYLIDINE

$C_6H_3(CH_3)_2(NH_2)$. Mol. Wt. 121.12.

A yellow to yellowish-brown liquid, having a specific gravity of 0.981 to 0.984, and boiling at 212 to 215° C.

TEST OF PURITY

Substances Insoluble in Hydrochloric Acid. — 10 cc. of xylidine should afford a perfectly clear solution with a mixture of 10 cc. of hydrochloric acid and 10 cc. of water.

ZINC

Zn. Atomic Wt. 65.4.

A shining, white metal, exhibiting a slight, bluish-gray luster, and occurring on the market in various forms.

I

ZINC, FREE FROM ARSENIC, SULPHUR, PHOSPHORUS, AND IRON

This grade of zinc occurs in three forms, — granulated, thin sticks, and thick sticks.

TESTS OF PURITY

Arsenic. — Introduce 20 gm. of zinc into the generating flask of a Marsh apparatus, and start the hydrogen with arsenic-free, dilute sulphuric acid (1:5), maintaining the flow of gas until the metal is almost completely dissolved. At the end of the test, no deposit of arsenic should be visible within the reduction tube.

Matter Oxidizable by Potassium Permanganate. — Dissolve 10 gm. of zinc in a mixture of 60 cc. of water and 15 cc. of pure sulphuric acid (sp. gr. 1.84) in a flask provided with a rubber valve; no black flocks should remain undissolved.

As soon as the zinc has dissolved, add to the solution, drop by drop, decinormal potassium permanganate. Not more than 0.1 cc. should be required to produce a distinct pink color. Should more of the permanganate solution be required, the dilute sulphuric acid (15 cc. of sulphuric acid and 60 cc. of water) should be titrated for the purpose of control, without zinc, using the same solution of decinormal potassium permanganate.

Compounds of Sulphur, Phosphorus, etc. — Into a narrow test-tube introduce 1 gm. of zinc together with 5 to 10 cc. of dilute, arsenic-free sulphuric acid, and in the upper part of the tube insert a plug of cotton, which serves to hold back the water carried off by the escaping hydrogen. Over the mouth of the tube lay a small piece of filter paper which has been moistened with a 1:1 silver nitrate solution, and carefully dried. The reaction is allowed to proceed in a dark place which is perfectly free from hydrogen sulphide. On standing two hours, the silver nitrate paper should show neither a yellow nor a black color.

II

ZINC, FREE FROM ARSENIC, NEARLY FREE FROM IRON

This grade of zinc is in various forms, — granulated, thick sticks, thin sticks, sheets.

TESTS OF PURITY

Arsenic. — Introduce 20 gm. of zinc into the generating flask of a Marsh apparatus, and start the hydrogen with arsenic-free, dilute sulphuric acid (1:5), maintaining the flow of gas until the metal is completely dissolved. At the end of the test no deposit of arsenic should be visible within the reduction tube.

Matter Oxidizable by Permanganate. Dissolve 10 gm. of zinc in a mixture of 60 *cc.* of water and 15 cc. of pure concentrated sulphuric acid in a flask provided with a rubber valve. The solution should contain only a very slight quantity of undissolved black flocks. As soon as the zinc has dissolved, add to the solution, drop by drop, normal potassium permanganate. Not more than 0.1 cc. should be required to produce a distinct pink color.

This grade of zinc *in powder form* should, in addition to the above, be tested as follows:

Chlorides. Dissolve 5 gm. of the zinc in a mixture of 50 cc. of nitric acid and 100 cc. of water. This solution should not show more than a slight opalescent turbidity on the addition of silver nitrate solution.

III

ZINC, FREE FROM ARSENIC

This zinc is used in the following forms: Granulated, thick sticks, thin sticks.

TEST OF PURITY

Arsenic. Introduce 20 gm. of zinc into the generating flask of a Marsh apparatus, and start the hydrogen with arsenic-free, dilute sulphuric acid (1 : 5), maintaining the flow of gas until the metal is almost completely dissolved. At the end of the reaction, no deposit of arsenic should be visible within the reduction tube.

IV

ZINC DUST

A fine, gray powder, containing about 90 per cent of metallic zinc.

TESTS OF PURITY

Nitrogen (from Nitrates and Ammonia). — Dissolve 10 gm. of zinc dust in a mixture of 20 cc. of sulphuric acid (sp. gr. 1.84) and 200 cc. of water. Add to the solution 100 cc. of sodium hydroxide solution I, distil off about 50 cc., and collect the distillate in a receiver containing about 20 cc. of water and 2 to 3 cc. of decinormal hydrochloric acid. Titrate the distillate with decinormal potassium hydroxide, using methyl orange as the indicator. Not more than 0.2 cc. of the acid should have been consumed by the ammonia.

Determining the Value of Zinc Dust. — Introduce 1 gm. of zinc dust together with a few glass beads into a flask of about 200 cc. capacity, closed with a glass stopper, and then add a mixture of 30 cc. of potassium iodate solution and 100 cc. of sodium hydroxide solution (potassium iodate solution: 15.25 gm. KIO_3 to 300 cc. H_2O; sodium hydroxide solution: 300 gm. NaOH to 1000 cc. H_2O). Now shake the contents of the flask vigorously for five minutes; transfer, without filtering, to a graduated flask of 1000 cc. capacity; fill up to the mark and mix. Of this solution transfer 100 cc. into an iodine distillation apparatus. Add 50 cc. of dilute sulphuric acid, and after expelling the air by means of carbon dioxide, distil, and receive the iodine in potassium iodide solution (1:5). The distillation is ordinarily complete in about twenty minutes, and may be recognized by the contents of the retort having become colorless. The iodine distilled over is titrated with decinormal sodium thiosulphate.

$6I = 15\ Zn$.*

1 cc. of decinormal $Na_2S_2O_3$ = 0.01635 gm. of Zn, log. 21352.

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* $15\ Zn + 30\ NaOH = 15\ Zn(ONa)_2 + 15\ H_2$.
$5KIO_3 + 15\ H_2 = 5KI + 15H_2O$.
$KIO_3 + 5\ KI + 3\ H_2SO_4 = 3\ K_2SO_4 + 3\ H_2O + 6I$.

ZINC CHLORIDE

$ZnCl_2$. Mol. Wt. 136.3.

A dry, white powder, deliquescent in the air, and easily soluble in water and in alcohol. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Solubility (Basic Zinc Chloride). The solution of 1 gm. of zinc chloride in 1 cc. of water should be clear, or at most only slightly turbid; any turbidity caused in the solution by the addition of 3 cc. of alcohol should disappear on the addition of 1 drop of hydrochloric acid.

Sulphates. — 10 cc. of the 1 : 10 aqueous solution acidulated with a few drops of hydrochloric acid should not be rendered turbid by barium chloride solution.

Foreign Metals and Alkalies.

(*a*) The solution of 1 gm. of zinc chloride in 10 cc of water acidulated with 1 cc. of hydrochloric acid should appear unchanged after the addition of hydrogen sulphide water.

(*b*) 1 gm. of zinc chloride should afford a clear solution with 10 cc. of water and 10 cc. of ammonia water. On adding hydrogen sulphide water to the solution, a pure white precipitate should form. On filtering, evaporating the filtrate to dryness, and igniting, no weighable residue should remain.

ZINC IODIDE STARCH SOLUTION

A colorless, clear, or only slightly opalescent liquid. It deteriorates with age, acquiring a blue color.

TESTS OF SENSITIVENESS

A mixture of 1 cc. of zinc iodide-starch solution and 20 cc.

of water should acquire an intense blue color on adding 1 drop of decinormal iodine solution.

Zinc iodide-starch solution diluted with 50 volumes of water should not acquire a blue color on the addition of dilute sulphuric acid.

ZINC OXIDE

ZnO. Mol. Wt. 81.4.

A white, amorphous powder, having a faint yellowish tinge. Zinc oxide is insoluble in water, but is soluble in dilute acetic acid and in mineral acids.

TESTS OF PURITY

Arsenic. A mixture of 1 gm. of zinc oxide and 3 cc. of stannous chloride solution should not acquire a darker color on standing one hour.

Sulphates and Chlorides. — On shaking 2 gm. of zinc oxide with 20 cc. of water and filtering, the acidulated filtrate should show no reaction on the addition of barium nitrate solution, and should acquire at most a slight opalescent turbidity on the addition of silver nitrate solution.

Carbonates, Calcium, Magnesium, and Foreign Heavy Metals. 1 gm. of zinc oxide should dissolve completely in 10 cc. of dilute acetic acid without effervescence. On adding to this solution 20 cc. of ammonia water, a clear colorless liquid should result which should not show a change on the addition of ammonium oxalate and sodium phosphate solutions, and which should give a pure white precipitate with hydrogen sulphide gas.

Foreign Bodies which Reduce Permanganate when Zinc Oxide is Dissolved in Dilute Sulphuric Acid. — Very carefully triturate 3 gm. of zinc oxide in a mortar with 20 cc. of water containing in solution 0.2 gm. of pure ferric alum free from ferrous salt. Then add to the mixture 25 cc. of diluted sulphuric acid, and effect complete solution by gently heating.

Dilute the solution with 100 cc. of thoroughly boiled and cooled water, and add decinormal potassium permanganate.

A single drop of the permanganate should suffice to impart a distinct pink color to the solution.

ZINC SULPHATE

$ZnSO_4 + 7 H_2O$. Mol. Wt. 287.57.

Colorless crystals, slowly efflorescent in dry air, soluble in 0.6 part of water, and insoluble in alcohol. The aqueous solution is acid to litmus paper.

TESTS OF PURITY

Chlorides. — The 1:20 aqueous solution should not be affected by silver nitrate solution.

Foreign Metals. — A solution of 0.5 gm. of zinc sulphate in 10 cc. of water and 5 cc. of ammonia water should be clear and should afford a pure white precipitate with hydrogen sulphide water.

Iron. — 1 gm. of zinc sulphate dissolved in 20 cc. of water acidulated with a few drops of nitric acid and boiled should not be reddened by potassium sulphocyanate solution.

Nitrates. — On adding to 5 cc. of a 1:10 zinc sulphate solution 1 drop of indigo solution and 5 cc. of concentrated sulphuric acid, the blue color should not disappear.

Ammonium Compounds. — On heating 1 gm. of zinc sulphate with sodium hydroxide solution, no vapors of ammonia should be evolved (to be ascertained by moist litmus paper).

Free Sulphuric Acid. — On shaking 2 gm. of zinc sulphate with 10 cc. of 85 per cent alcohol and, after ten minutes, filtering, the filtrate, when diluted with 10 cc. of water, should not redden blue litmus paper.

Arsenic. — Introduce 20 gm. of arsenic-free, metallic zinc into the generating flask of a Marsh apparatus, and start the hydrogen with dilute (1:5) sulphuric acid. Dissolve 2 gm. of zinc sulphate in 20 cc. of water, introduce the solution in small quantities at a time into the Marsh apparatus, and maintain a slow stream of gas for about half an hour. At the end of this period no deposit of arsenic should be visible within the reduction tube.

INTERNATIONAL ATOMIC WEIGHTS, 1906

Aluminum	Al	27.1	Hydrogen	H	1.008	Rubidium	Rb	85.5
Antimony	Sb	120.2	Indium	In	115	Ruthenium	Ru	101.7
Argon	A	39.9	Iodine	I	126.97	Samarium	Sm	150.3
Arsenic	As	75.0	Iridium	Ir	193.0	Scandium	Sc	44.1
Barium	Ba	137.4	Iron	Fe	55.9	Selenium	Se	79.2
Bismuth	Bi	208.5	Krypton	Kr	81.8	Silicon	Si	28.4
Boron	B	11	Lanthanum	La	138.9	Silver	Ag	107.93
Bromine	Br	79.96	Lead	Pb	206.9	Sodium	Na	23.05
Cadmium	Cd	112.4	Lithium	Li	7.03	Strontium	Sr	87.6
Cæsium	Cs	132.9	Magnesium	Mg	24.36	Sulphur	S	32.06
Calcium	Ca	40.1	Manganese	Mn	55.0	Tantalum	Ta	183
Carbon	C	12.0	Mercury	Hg	200.0	Tellurium	Te	127.6
Cerium	Ce	140.25	Molybdenum	Mo	96.0	Terbium	Tb	160
Chlorine	Cl	35.45	Neodymium	Nd	143.6	Thallium	Tl	204.1
Chromium	Cr	52.1	Neon	Ne	20	Thorium	Th	232.5
Cobalt	Co	59.0	Nickel	Ni	58.7	Thulium	Tm	171
Columbium	Cb	94	Nitrogen	N	14.04	Tin	Sn	119.0
Copper	Cu	63.6	Osmium	Os	191	Titanium	Ti	48.1
Erbium	Er	166	Oxygen	O	16.00	Tungsten	W	184
Fluorine	F	19	Palladium	Pd	106.5	Uranium	U	238.5
Gadolinium	Gd	156	Phosphorus	P	31.0	Vanadium	V	51.2
Gallium	Ga	70	Platinum	Pt	194.8	Xenon	Xe	128
Germanium	Ge	72.5	Potassium	K	39.15	Ytterbium	Yb	173.0
Glucinum	Gl	9.1	Praseodymium	Pr	140.5	Yttrium	Yt	89.0
Gold	Au	197.2	Radium	Rd	225	Zinc	Zn	65.4
Helium	He	4	Rhodium	Rh	103.0	Zirconium	Zr	90.6

1 CC. OF NORMAL HYDROCHLORIC ACID IS THE EQUIVALENT OF:

	Gram.	Log.
Ammonia Water NH_3	0.01706	23198
Barium Hydroxide $Ba(OH)_2+8\ H_2O$	0.15777	19802
Calcium Hydroxide $Ca(OH)_2$	0.03705	56879
Potassium Bicarbonate $KHCO_3$	0.10015	00065
Potassium Carbonate K_2CO_3	0.06915	83979
Potassium Hydroxide KOH	0.5615	74935
Sodium Bicarbonate $NaHCO_3$	0.08405	92454
Sodium Borate, Anhydrous* $Na_2B_4O_7$	0.10105	00453
Sodium Borate, Cryst.* $Na_2B_4O_7+10\ H_2O$	0.19113	28132
Sodium Carbonate, Anhydrous Na_2CO_3	0.05305	72469
Sodium Carbonate, Cryst. $Na_2CO_3+10\ H_2O$	0.14313	15573
Sodium Hydroxide NaOH	0.04005	60260

* Indicator: Methyl Orange.

1 CC. OF DECINORMAL POTASSIUM PERMANGANATE SOLUTION IS THE EQUIVALENT OF:

	Gram.	Log.
Acid Oxalic, Anhydrous $H_2C_2O_4$	0.0045008	65329
Acid Oxalic, Cryst. $H_2C_2O_4 + 2\,H_2O$	0.0063024	79951
Ammonium Oxalate $(NH_4)_2C_2O_4 + H_2O$	0.007108	85175
Hydrogen Peroxide H_2O_2	0.0017008	23065
Indigotin (Indigo Blue) $C_{17}H_{10}N_2O_2$	0.007415	87011
Iron Fe	0.00559	74741
Iron Sulphate, Ferrous $FeSO_4 + 7\,H_2O$	0.027807	44415
Iron and Ammonium Sulphate $FeSO_4(NH_4)_2SO_4 + 6\,H_2O$	0.039226	59358
Potassium Nitrite KNO_2	0.0042595	62936
Potassium Oxalate, Neutral $K_2C_2O_4 + H_2O$	0.0092158	96453
Potassium Tetraoxalate $KHC_2O_4 + H_2C_2O_4 + 2\,H_2O$	0.006355	80312
Silver Nitrite $AgNO_2$	0.0076985	88640
Sodium Nitrite $NaNO_2$	0.0034545	53838
Sodium Oxalate $Na_2C_2O_4$	0.006705	82640

1 CC. OF NORMAL POTASSIUM HYDROXIDE SOLUTION IS THE EQUIVALENT OF:

	Gram.	Log.
Acid Acetic $HC_2H_3O_2$	0.06003	77837
Acid Acetic Anhydride $(CH_3{\cdot}CO)_2O$	0.051024	70777
Acid Citric $C_6H_8O_7 + H_2O$	0.07002	84522
Acid Hydriodic HI	0.12797	10710
Acid Hydrobromic HBr	0.08096	90827
Acid Hydrochloric HCl	0.036458	56179
Acid Hydrofluoric HF	0.020	30103
Acid Nitric HNO_3	0.06304	79962
Acid Oxalic, Cryst. $H_2C_2O_4 + 2\,H_2O$	0.063024	79950
Acid Oxalic, Sublimed $H_2C_2O_4$	0.045008	65329
Acid Phosphoric *H_3PO_4	0.09802	99131
Acid Phosphoric † H_3PO_4	0.04901	69028
Acid Succinic $C_4H_6O_4$	0.05902	77100
Acid Sulphuric H_2SO_4	0.049038	69053
Acid Sulphuric Anhydride SO_3	0.04003	60239
Acid Tartaric $C_4H_6O_6$	0.07502	87518
Hydroxylamine Hydrochloride † $NH_2OH{\cdot}HCL$	0.06952	84211
Potassium Bisulphate $KHSO_4$	0.13621	13421
Sodium Bisulphate $NaHSO_4$	0.13813	14029

* Indicator: Methyl Orange. † Indicator: Phenolphthalein.

1 CC. OF DECINORMAL SODIUM THIOSULPHATE SOLUTION IS
EQUIVALENT OF:

	Gram.
Acid Iodic HIO_3	0.002933
Acid Iodic Anhydride I_2O_5	0.0027828
Barium Peroxide BaO_2	0.00847
Bromine Br	0.007996
Chlorine Cl	0.003545
Chromium Trioxide CrO_3	0.003336
Hydrogen Peroxide H_2O_2	0.0017008
Iodine I	0.012697
Iron Fe	0.00559
Potassium Biniodate $KIO_3 \cdot HIO_3$	0.00325082
Potassium Bromate $KBrO_3$	0.00278516
Potassium Chromate K_2CrO_4	0.00648
Potassium Dichromate $K_2Cr_2O_7$	0.0049083
Potassium Iodate KIO_3	0.0035686
Sodium Bromate $NaBrO_3$	0.002517

1 CC. OF DECINORMAL IODINE SOLUTION IS THE EQUIVALENT

	Gram.
Acid Arsenous As_2O_3	0.00495
Acid Sulphurous SO_2	0.003203
Iron Sulphide FeS	0.004398
Potassium Bisulphite $KHSO_3$	0.0060105
Sodium Bisulphite $NaHSO_3$	0.0052059
Sodium Sulphide $Na_2S + 9\,H_2O$	0.0120152
Sodium Sulphite, Cryst. $Na_2SO_3 + 7\,H_2O$	0.0126136
Sodium Sulphite, Anhydrous Na_2SO_3	0.006308
Sodium Thiosulphate $Na_2S_2O_3 + 5\,H_2O$	0.02483
Stannous Chloride $SnCl_2 + 2\,H_2O$	0.0112966

1 CC. OF FIFTH NORMAL HYDROCHLORIC ACID IS THE EQUIVALENT OF:

	Gram.	Log.
Sodium Borate Cryst.* $Na_2B_4O_7 + 10H_2O$	0.038226	58235
Sodium Borate, Anhydrous *$Na_2B_4O_7$	0.02021	30557
Sodium Oxalate (after ignition) $Na_2C_2O_4$	0.01341	12743

* Indicator: Methyl Orange.

1 CC. OF FIFTH-NORMAL POTASSIUM HYDROXIDE SOLUTION IS THE EQUIVALENT OF:

	Gram.	Log.
Acid Oxalic, Cryst. $H_2C_2O_4 + 2H_2O$	0.012605	10055
Acid Oxalic, Anhydrous $H_2C_2O_4$	0.009001	95432
Potassium Biniodate * $KIO_3{\cdot}HIO_3$	0.078018	89219
Potassium Tetraoxalate *$KHC_2O_4 + H_2C_2O_4 + 2H_2O$ ..	0.016947	22909

* Indicator: Phenolphthalein.

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